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CURRENT DEVELOPMENTS IN FIBER-REINFORCED COMPOSITES

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FOREWORD

This report was initiated in the Air Force Materials Laboratory under Project No. 7351, "Metallic Materials," Task No. 735107, "Metal Matrix Composites." It was released by the authors 1 October 1967 for publication as a technical report.

This report was coauthored by Capt James A. Snide, and Dr. C. T. Lynch of the Advanced Metallurgical Studies Branch, Metals and Ceramics Division, and Lt Col L. D. Whipple of the Advanced Composites Division, Air Force Materials Laboratory, Directorate of Laboratories, Wright-Patterson Air Force Base, Ohio. It covers a study conducted from January 1967 to May 1967.

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This technical report has been reviewed and is approved.

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ABSTRACT

A review of recent progress in composite technology is presented with primary emphasis on fiber-reinforced metal-matrix composites. A brief discussion of the development of high strength, high modulus, low density filaments is included. Current efforts on plastic and ceramic matrix composites have been considered for comparative purposes. Filament-matrix compatibility has been identified as the major limiting factor in high temperature fabrication and utilization of metal-matrix composites. Some approaches to the solution of this compatibility problem are discussed. The various fabrication methods have been reviewed and the mechanical behavior of metal-matrix composites is illustrated with a boron-reinforced aluminum system. The method taken to expedite the development of advanced composites as practical engineering structures is discussed. This approach has been to integrate the efforts of materials engineers, designers, and fabricators into a single team.

This team concept is illustrated by showing the progress of the development of fiber-reinforced plastic aerospace structures. In gas turbine engine applications, the need for increased temperature capability must be met with metal-matrix composites. Fabrication requirements show a definite need for automated tape layup techniques. Hand-layup techniques could not provide cost effective procedures for production of operational systems hardware. Current program efforts indicate that theoretically weight can be reduced by using composite structures. Sometimes in practical applications even greater weight savings can be realized.

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SECTION I

INTRODUCTION

An increase in effort in composite technology has been noted in the last few years mainly as a result of (1) the discovery and development of high strength, high modulus fibers and (2) the demonstration that a metal matrix can provide sufficient shear strength so that even short whisker filaments can bear an appreciable fraction of a tensile load applied to a composite (Reference 1). The concept of reinforcing a material by the use of a fiber is not a new one. The Egyptian bricklayer employed the same principle more than three thousand years ago when straw was incorporated into the bricks. More recent examples of fiber reinforced composites are steel reinforced concrete, nylon and rayon reinforced tires, and fiberglass reinforced plastics.

The Air Force is interested in the development of composite structures to meet the need for high performance materials for new systems. Composites appear to offer the following potential advantages:

(1) Improved materials properties

(2) Flexibility in design

In aerospace applications, lighter, stronger, stiffer, and higher temperature materials are constantly being sought. Figure 1 is a schematic which is intended to represent the current state-of-the-art of materials technology. This curve defines a "materials technology envelope" from which the designer may select materials for aerospace application. The specific materials properties indicated on the ordinate of this curve include strength, modulus, fracture toughness, stress-rupture, creep rate, resistance to oxidation, etc. If weight is a major constraint in application, the materials selected would probably be selected in order from left to right with increasing temperature. The shaded areas show the improved properties that can be expected from nominal improvements in alloy composition, processing, and design, arising from the normal competitive discourse of the materials industry. An approach which offers the opportunity of significantly improved material properties with a resultant shift of this materials technology envelope upward and to the right, is to use materials having low-density, high-strength, and high-modulus as reinforcements. The benefits are obvious. With reduced weight, a structure will be more efficient and a vehicle will have longer range or greater payload. If materials can operate at current stress levels but at higher temperatures, propulsion systems will have greater efficiencies which will yield better systems performance.

Composite materials offer design flexibility which enables the designer to use the material more effectively. The internally pressurized cyclinder is a simple example of the more effective use of material. In this case, the hoop stress is twice the longitudinal stress. If an isotropic material is used, the cylinder would be over-designed by twice the material thickness in the longitudinal direction of the cylinder. Conceptually then with composites, balanced design may be employed which will yield further weight reduction. The key point is that the material can be designed to accommodate the defined stress distribution.

The emphasis of this paper is directed toward the development of metal-matrix composites; however, it is appropriate to briefly consider the status of plastic and ceramic matrix composites which compete with metallic matrix composites for increased research and development emphasis.

SECTION II

PLASTIC-MATRIX COMPOSITES

The Air Force Materials Laboratory is currently directing plastic composites research in the following three general thermal regimes (Reference 2):

(1) Near ambient temperatures, with maximum temperature, peaks of 300°F or less. Applications include subsonic aircraft structural components, rocket motor cases, and engine compressor blades.

(2) Long-term capability at 600°F. This temperature results from aerodynamic heating at Mach 3 flight.

(3) Temperatures above 600°F. Requirements result from Mach 3+ flight, higher performance engines, and aerospace vehicles.

Research is proceeding along a broad front on the reinforcements, matrix, coupling agents, composite fabrication, micromechanics, and composite evaluation. The increased requirements for high temperature stability beyond the range of traditional plastic matrices has led to new plastic matrix materials such as epoxy, phenolic, polyimide, and polybenzimidazole resins. Illustrations of the advanced development of plastics composites will be given subsequently.

SECTION III

CERAMIC-MATRIX COMPOSITES

Research on ceramic-matrix composites has been conducted for the last ten years (References 3 through 7) using metal fiber reinforcements. These studies indicate that ceramic matrices containing short length (1/8 to 1/2 in.), small diameter (2 to 10 mil) filaments evidenced substantially improved thermal shock resistance compared to the ceramic without reinforcement. This seems to be true whether the system is one which exhibits little cracking because of a compatible differential thermal expansion of the fiber and matrix, or an incompatible differential thermal expansion which results in formation of a crack system. In a compatible system, the coefficient of thermal expansion of the matrix is equal to or exceeds that of the fiber. In an incompatible system, the coefficient of expansion is greater for the matrix than for the fiber. For most ceramic materials with refractory metal wire reinforcements, the coefficient of thermal expansion of the matrix ceramic exceeds that of the fiber. The ceramic is placed in tension through high temperature processing, thus producing severe matrix cracking. Nevertheless, ceramic composites have exhibited mechanical failure at predictable locations, and stress-strain diagrams have shown that the reinforcement serves to hold the composite together after the ceramic itself has failed. Thus it is possible to eliminate catastrophic brittle fracture, and research efforts continue in an effort to find practical systems for further development (References 8 and 9).

Figure 2 (Reference 5) shows the stress-strain behavior of a clay-feldspar ceramic reinforced with tungsten fibers. On successive loadings, the specimen is permanently deformed, and finally fails under conditions beyond that where the ceramic itself has failed. Close examination of a specimen showed that first the ceramic fails at approximately the same value stress level as an unreinforced specimen, and then the outer fibers are gradually pulled loose from the ceramic matrix. Finally, failure occurs when the outer fibers reach a stress sufficient to completely separate them from the ceramic matrix.

Model systems have been designed in which the thermal expansion of the matrix matches the thermal expansion of the fiber. Results of matrix properties for such a body consisting of mullite and alumina reinforced with molybdenum fiber are listed in Table I. A significant improvement of properties, as a result of the fiber assuming a proportional amount of the stress is noted. In Figure 3, the modulus of rupture as a function of thermal cycling for this mullite-alumina-molybdenum fiber system (Body 712) is shown (Reference 4). An unreinforced body has an insignificant modulus value after almost two cycles. Very low volume percent molybdenum reinforcement is equally unsatisfactory.

Because of the differential thermal expansion problem with most systems of interest at high temperatures, the potential for reinforced ceramic-matrix composites is inherently limited. Some of the limitations are disappearing with the development of new filaments. For example, high quality alumina whiskers offer the potential of reinforcing alumina with alumina. New reinforcements may serve to broaden the interest in ceramic-matrix composite systems. The reinforcement-matrix compatibility and the retention of reinforcement properties during processing at high temperatures and pressure are the major problems. Low temperature forming operations may provide one solution to these problems. Rapid hot pressing, limiting destructive interaction, may offer another solution to minimizing reinforcement-matrix interactions. As these pacing problems are solved, ceramic matrix composites may emerge as candidate composite materials for refractory, protective, and other applications.

SECTION IV

METAL-MATRIX COMPOSITES —> 5

1. POTENTIAL ADVANTAGES

The most obvious potential advantage of a metal matrix is in resistance to long-term exposure to many severe environments. Metals have a better resistance to erosion and to chemical attack at high temperatures than the various plastics which are used as matrix materials. The strength retention of currently available reinforcements such as boron, SiC, and Al_2O_3 offer the potential for high temperature composites by combining these reinforcements with the appropriate matrices. Specific comparisons must depend on the nature of the application, the loading, the time at temperature, and other factors, but it is reasonable to assume that metal matrices may provide significant competition to polymer matrices at temperatures above 250°F. Very significant advances in temperature resistant polymers are anticipated (Reference 10) and will influence the comparison, but as recent analyses indicate (Reference 10), there is no question that metal matrices are unchallenged at higher temperatures.

Other possible advantages of metal matrices can be illustrated by considering some of the factors which currently prevent the use of unidirectional strength-to-weight ratio of filaments as a direct indication of fabricated weight of a structural element. Table II is derived from a recent study (Reference 11) of the optimization of glass filament-wound solid-propellant rocket-motor cases. For specific application, the weight of optimized filament-wound cases is compared with cases fabricated from metals with the design allowables shown. Note that the enormous advantages of the strength-to-density ratio of the composites over metals is not reflected in a comparable decrease in the weights of the final cases. Some of the reasons for this are as follows:

(1) The composite is, of course, not 100% filament. These particular examples contained 18.8 weight percent epoxy. Although the polymer matrix performs a necessary function in permitting the strength of the filaments to be utilized, it is generally assumed that the matrix does not itself provide a significant contribution to either the strength or modulus of the composite.

(2) The filaments are assumed to be effective only along their axes. If the application involves a multiaxial stress field, additional filaments must be added to bear the load.

(3) Additional reinforcement must be added to the filament-wound structures to cope with stiffness requirements and for cutouts, attachments, and other stress concentrations, that is, at any point where stresses occur transverse to the filaments or shear loads, or where the continuity of the filaments is disturbed, or both.

Metal matrices offer several possible advantages to offset possible disadvantages of density and more difficult fabrication. A metal can offer significantly greater tensile, shear, bearing, and transverse strength as well as greater deformation prior to fracture than current polymer matrices. The matrix can provide a meaningful contribution to the elastic strength and modulus of the composite in all directions. The match of modulus of the reinforcement and the matrix has been shown to reduce the stress concentration within the composite (Reference 12). Figure 4 shows the advantage of similar reinforcement and matrix modulus when circular filaments in a square array are subjected to longitudinal shear loading. As G_f/G_m approaches one, the stress concentration approaches one. Also, if the mechanism by which the composite

fractures involves a statistical accumulation of individual fiber breaks, a metal matrix may delay the onset of final catastrophic failure and thus increase the strength.

The principal steps necessary for the development of metal matrix composites are shown in Figure 5. This program requires independent emphasis on each step with consideration of the interdependence of each step with each other. Emphasis is made to combine compatible systems of the reinforcement and the matrix by selected fabrication methods and to evaluate the resultant composite properties. Simultaneously efforts are made to develop an understanding of composite behavior, an ability to predict this behavior, and to develop nondestructive test techniques. The role of the constituents in fiber composites is shown in Figure 6 (Reference 13). In comparing Figures 5 and 6 the development effort simply parallels the role of the constituents in fiber-reinforced composites.

2. REINFORCEMENTS

The first and most widely used reinforcing fiber is formed from glass. The reinforcement of structural plastics until the last few years consisted primarily of "E" glass fibers having a modulus of 10.5×10^6 psi and tensile strength of 400×10^3 psi and more recently has been "S" glass having a modulus of 12.5×10^6 psi and tensile strength of 600×10^3 psi.

While the glass fibers were being widely utilized, a broad spectrum of unique fibers were being developed by the Air Force Materials Laboratory to attain greater modulus, higher tensile strength, and lower density. One of these fibers was boron which is formed by vapor deposition on a tungsten substrate. Deposition occurs when a hot tungsten wire (approx. 1000°C) is passed through a vaporization chamber containing boron trichloride and hydrogen. The typical boron deposit is a 4-mil diameter on a 0.5-mil substrate. Typical tensile strength of this filament is 460×10^3 psi with a standard deviation of 92×10^3 psi. This filament has a modulus of elasticity (Young's modulus) of 60×10^6 psi, which has been measured using a long gauge length to minimize experimental errors. A fatigue life, in excess of a million cycles (using a tension-zero-tension cycle at 150 cycles per minute), has been measured using a cyclic load of half the mean tensile strength. The density of 2.6 gm/cm^3 is slightly higher than "E" glass, but the specific modulus of the boron is superior to that of glass fiber (Reference 4).

Substrates other than tungsten are being examined to reduce the cost and to produce a boron filament having a density closer to the bulk density of 2.34 gm/cm^3 boron. The use of fused silica as the substrate is an example. Diborane is broken down by chemical decomposition at 700°C to form a boron coating on the silica. The lower deposition temperature for diborane permits the substitution of silica for the tungsten stubstrate. This route may reduce costs by employing a cheaper substrate than tungsten and eliminating reactor shutdown due to the finite length of commercial tungsten wire. The silica-boron filament has tensile strength of 350×10^3 psi and modulus of elasticity of 59×10^6 psi and a density of 2.35 gm/cm^3 (Reference 15).

Silicon carbide has been deposited on a tungsten substrate using ethyltrichlorosilane, hydrogen, and argon (Reference 16). Although silicon carbide has a higher density than boron (3.4 gm/cm) the modulus of elasticity of 71×10^6 psi and tensile strength of 340×10^3 psi are sufficiently high to encourage further studies of this fiber as a reinforcement. There are many other inorganic species amenable to fiber formations by chemical deposition. These include boron carbide, titanium diboride, and boron silicides.

Considerable effort has been expended in recent years to produce high strength graphite fibers. Fiber synthesis usually involves pyrolysis and high-temperature processing of polymeric precursor materials. Graphite yarn is commercially available which exhibits approximately 200 x 10^3 psi tensile strength, 25 x 10^6 psi elastic modulus, and a density of 1.4 gm/cc. The promise of excellent mechanical properties-to-weight ratio makes graphite an extremely attractive candidate for reinforcement of structural and ablative composite materials. Exploratory development is currently concentrating on selection of precursor materials and processing parameters (Reference 17).

Some properties of metal wires, vapor-deposited filaments, and whiskers, which are being used in metal-matrix composites, are listed in Table III. The relative sizes of these different reinforcements are indicated in Figure 7 (Reference 13).

3. REINFORCEMENT-MATRIX COMPATIBILITY PROBLEM

The pacing problem in the development of metal-matrix composites for high-temperature application is to control the filament-matrix compatibility during composite fabrication and to control it at the intended use-temperature. Several approaches are being pursued to control the filament-matrix compatibility:

(1) develop new reinforcements which are thermodynamically stable with respect to the matrix;

(2) develop diffusion-barrier coatings which reduce the filament-matrix interaction;

(3) develop alloy additions which reduce the activity of the diffusing species and enhance the physical compatibility, such as, minimizing the thermal expansion mismatch at the interface.

These approaches offer a solution to the problem through control of the matrix-filament interface.

Various types of filament-matrix interactions have been reported (References 18 and 19). There are two types of related interface reactions which should be emphasized. First, the nature of the critical interfacial reaction may not be obvious. Illustratively, the TiB_2 reaction zone, which is formed during composite fabrication and subsequent thermal treatment of the boron-Ti-8Al-1Mo-IV, is shown in Figure 8. The void in the unreacted boron is due to Kirkendall porosity which is caused by the predominantly one-way diffusion of boron. This is evidenced by the lack of recession of the original boron filament interface. The void area adjacent to the tungsten core has recently been shown (Reference 20) to be conchoidal fractures which occur during polishing, probably as a result of the complex stress state due to the tungsten-boron interaction. Herzog (Reference 18) has described formation of the radial fissures as also due to the residual stress caused by tungsten boride formation in filament deposition. A more subtle change during the reaction is shown in Figure 9 of the electron microprobe trace across the filament, the interface, and into the matrix. Notice the increased aluminum concentrations in the matrix immediately adjacent to the TiB_2 layer. This is due to the rejection of aluminum in the formation of the TiB_2 reaction layer. This high aluminum concentration in the matrix may embrittle the matrix or change the stability of the various phases in the alloy.

The second point can be illustrated by the Al-B system. Figure 10 is a photomicrograph of an aluminum-boron composite prepared by solid state bonding which shows that interfacial

reaction has occurred. Figure 11 shows the electron microprobe X-ray intensity and specimen current of boron and aluminum, which further substantiates the lack of interfacial reaction (Reference 21). In contrast is the reactivity evidenced in an aluminum-boron composite prepared by liquid infiltration in which the boron filament is in contact with molten aluminum at 1350°F for approximately 30 seconds (Reference 22). The filament-matrix interaction is quite evident in Figure 12. The Al-B phase diagram predicts these results. Precise control of the time in which boron is in contact with molten aluminum, however, permits the fabrication of successful composite structures. Figure 13 (Reference 22) is a photomicrograph of an aluminum-boron composite prepared so that the boron was in contact with liquid aluminum for less than three seconds. Notice the significant reduction in the filament-matrix interaction. This points out that kinetic data is equally important as equilibrium data in the control of the interface in the composite.

It is possible, then, to use a thermodynamically unstable system at temperatures and for certain time periods in which the kinetics of the reaction processes provide minimal interaction. These reactions must not cause filament degradation or change in matrix properties. Thermodynamically unstable systems may be improved by matrix alloying and by interposing a diffusion barrier between the filament and the matrix. An alternate approach is to develop new reinforcements which will be more stable in the matrices of interest. For example, B_4C and SiC are stable in molten aluminum, and are therefore compatible reinforcements for aluminum alloys at high temperatures. The development of continuous Al_2O_3 filaments, which now appear feasible (Reference 23), will permit the reinforcement of higher temperature matrices.

4. FABRICATION TECHNIQUES

The various composite fabrication techniques which ~~will be~~ are briefly described are diffusion bonding, powder metallurgy, liquid infiltration, plasma spraying, electroforming, and unidirectional solidification. → 9

Two mechanized techniques, adaptable to preparing aligned filament mats, are filament winding and weaving. The apparatus by which filaments are wound on a mandrel mounted on a lathe is shown in Figure 14 (Reference 24). The loom, similar to that used for weaving cloth, is employed to prepare filament-metal ribbon mats as seen in Figure 15 (Reference 24). These processes are used to control the filament spacing during subsequent diffusion bonding. The weaving process is more complex because it introduces additional interfaces into the fabricated composites and the mechanical damage to filaments during weaving has not been evaluated. A typical diffusion-bonding process is illustrated schematically in Figure 16 (Reference 25). This process has been used to make composite sheet of several materials. The largest composite sheet made by a diffusion-bonding process to date has been a 25 volume percent AM 355 stainless steel reinforced 2024 Al matrix composite 8 feet long by 1 foot wide, by Harvey Aluminum.

One of the newest techniques for fabricating composites from metal powders is illustrated in Figure 17 (Reference 26), showing the composite billet for high-energy-rate-forming composite plates. This process has the advantage of relatively low temperature for very short times which prevents interaction of the reinforcement and matrix during fabrication. This high-energy process eliminates filament-matrix interaction but the effect on the reinforcement properties has not been evaluated. Also, the control of the reinforcement alignment, spacing, and distribution has not been solved although secondary processing will probably be used to align these short-fiber composites. Several whisker and chopped filament reinforced composites have been produced employing this technique although little mechanical data are available.

Figure 18 (Reference 27) is a typical glass system used in the laboratory to infiltrate liquid metal into pre-aligned reinforcements. This technique has not received wide use because of the few reinforcements which are stable in molten metals; however, this technique has been used to fabricate Al_2O_3 whisker reinforced composites. The problem with the Al_2O_3 whisker is not whisker-degradation reactions, but rather the proper wetting of the whisker with the matrix. The wetting is controlled by various coatings, matrix alloying, and control of the infiltration atmosphere.

Figure 19 (Reference 27) illustrates the device for plasma spraying the matrix while controlling the spacing of filament with a filament winding technique. The plasma spraying is performed in vacuum to prevent oxidation of the matrix. After spraying, the material is hot-pressed to eliminate the voids formed by shadowing behind the filaments in spraying. Although the matrix is molten when it is sprayed, little or no interaction has been found between boron and aluminum composites prepared by this method. The metal solidifies almost instantaneously when in contact with the filaments. Alloy matrices may also be prepared by this method. Surfaces of revolution may be similarly prepared although provisions must be made for the consolidation step after spraying.

A schematic of the electroforming process is shown in Figure 20 (Reference 28). The metal is deposited on a mandrel from a plating bath while the filaments are wound on the mandrel. This process offers the advantage that it may be accomplished at room temperature, eliminating filament matrix interaction. Boron-filament-reinforced nickel matrix composites have been fabricated using the electroforming method and the properties have been satisfactory. The boron-nickel system is of little practical interest because of the rapid interaction at high temperatures and the low melting eutectic temperature. The two practical limitations which seem to be inherent in the electroforming process are: (1) the impurities incorporated into the composite from the bath during deposition; and (2) inability to deposit alloys from solution.

Unidirectional solidification is an exception to the methods which have been presented. In this process, the reinforcing phase is grown in situ during controlled solidification from the melt (Reference 29). Fibrous or lamellar structures are produced depending upon the system, the composition of the system, and the rate of solidification, etc. In Figure 21 (Reference 30), the two types of morphology are shown. Unidirectional solidified composites have the advantage that the reinforcing phase is grown on low energy planes in equilibrium with the matrix metal (Reference 30). This provides high temperature stability for the phases because there is little driving force for reinforcement-matrix reactions. Salkind, et at (Reference 31), has shown that the $Al-Al_3N$ system is stable after 500 hours at 80% of the eutectic temperature. Salkind, et al (Reference 30) has shown that a turbine blade shape can be directionally solidified in the Ni-Mo system. In binary eutectic systems, the amount of reinforcing phase is dictated by the phase diagrams. In practical systems of interest, many have an insufficient amount of reinforcing phase to make them competitive with conventional alloys. Another approach to obtaining a larger amount of the reinforcing phase is to solidify a ternary alloy along the eutectic trough (Reference 32). This approach looks encouraging; however, initial attempts have been handicapped by the lack of appropriate phase diagrams.

5. MECHANICAL BEHAVIOR

Much of the current theory of the behavior of metal matrix composites is based on studies of metal-wire model systems and has been recently reviewed (References 33 and 34). Some results will be presented which illustrate the behavior of metal-matrix composites employing the higher-modulus filaments, that is, boron-reinforced-aluminum matrix systems. Generally, the tensile properties can be predicted by a linear rule of mixtures assuming equal strain in the matrix and filament. Deviation from this prediction can occur through poor fiber alignment,

poor bonding, fiber-matrix interaction during fabrication, and many other factors. Stuhrke (Reference 35) has recently shown the synergistic effect which is caused by factors related to the Poisson's ratio of the two components of the composite during load application.

The tensile strength of a plasma-sprayed aluminum-boron composite as a function of temperatures is shown in Figure 22 (Reference 36). The drop-off in tensile properties at higher volume-fraction reinforcement is attributed to mechanical damage of the filaments. Recent tests (Reference 32) have achieved a tensile strength of 185×10^3 psi at 55 V/O through process improvements. A comparison of the specific strength versus temperature for two composites and two conventional alloys is shown in Figure 23 (Reference 36). The boron-aluminum curve is quite flat indicative of its strength retention at elevated temperatures. Silica-reinforced aluminum loses its strength at a lower temperature, as would be expected. The comparison of composite properties with conventional alloys in Figure 23 may be misleading. The conventional alloys look unattractive when compared to the boron aluminum composite. It must be realized that the composites' properties are for unidirectional composites loaded in that direction. The cross-tension properties of the composite may be the same or lower than the matrix per se. Therefore, the indicated advantages in specific strength may be usable only if the composite is properly loaded. This may result in design limitations on the design or may require cross-plied material to handle complex loading which exceeds the unreinforced matrix strength.

The fatigue behavior of a metal-matrix composite is shown in Figure 24 (Reference 36). It can be seen that the endurance limit is at a higher stress level than the unreinforced aluminum. Figure 25 (Reference 36) shows the stress rupture behavior of the composite which is a significant improvement over the unreinforced matrix. Similar improvement in the creep behavior of a tungsten-reinforced Inconel 600 composite is shown in Figure 26 (Reference 13). The strain decreases markedly with increasing filament loading. For high filament content, a distinct secondary and tertiary creep is depicted.

Experimental results to date have indicated that the composite properties are dependent on the properties of both the reinforcement and the matrix with the reinforcement playing a more important role with increasing temperature. The interrelationship of the metallurgical and mechanical variables affecting composite behavior has not as yet been illucidated.

6. NONDESTRUCTIVE TESTING

The development of composite materials for structures applications necessitate inspection by some nondestructive test methods. New NDT techniques are being developed and old ones adapted to ensure the quality control of metal-matrix composites. Figure 27 (Reference 24) is a microradiograph of B-Ti composite after tensile testing. The breaks in the W-core of the boron filament are clearly visible. Similar radiographs of multilayered composites become quite difficult to analyze as the image of the various layers are superimposed. Five layers are about the maximum thickness which can be conveniently analyzed with the boron filament. This method cannot be used for a silica-core boron filament, since the x-rays resolve the tungsten core rather than the boron. In Figure 28, the capability of Ultrasonic C-Scan to detect disbonded areas in a diffusion bonded tungsten wire reinforced copper-matrix composite is illustrated (Reference 24). Similar results have been obtained in our laboratory on boron-reinforced aluminum sheet.

SECTION V

DESIGN AND FABRICATION OF AEROSPACE STRUCTURES

The scientific and engineering problems associated with research and development of composite materials have been reviewed. Some examples of design and fabrication of aerospace structures from advanced composites will not be presented. Although one metal-matrix composite system does show promise for further development and is currently being considered for a potential aerospace structures application, the technology of metal-matrix composites is still in its early stages when compared to the plastic-matrix composites. Therefore, it is appropriate to emphasize the advanced development of fiber-reinforced plastics where considerable effort has been in progress for several years.

On the basis of the state-of-the-art provided by exploratory development efforts and the potential payoffs projected in applications studies, objectives were developed for a US Air Force Advanced Development Program. The overall objective of the program is to conclusively demonstrate the high potential payoffs of application of advanced composites in aerospace structural and propulsion systems.

The approach taken to expedite the development of advanced composites was to integrate the efforts of materials engineers, designers, and fabricators into a single-team effort. It is this team concept that is currently being employed both in the Air Force management activities and in the participating industry activities. Too often in the past, new materials have been developed in the laboratory and have been placed "on-the-shelf" for use by designers who didn't understand the materials and fabricators who couldn't handle them. The purpose of the advanced composite materials, design, and fabrication team is to not only accelerate the advancement of the technology, but also to ensure that a trained, experienced high-confidence-level industry is ready to project the technology into usable, operational system hardware.

In October 1965, component hardware development efforts were initiated. These were in the application areas of aircraft structures, helicopter rotor blades, aerospace vehicles, and gas turbine engines. The purpose of these programs was to take a first, hard look at problems involved in designing, fabricating, and testing hardware using advanced composite materials. Another purpose, and perhaps even more important, was to provide meaningful direction to supporting materials development efforts.

Each of the component development efforts included program studies, technological base activities, and component development. Program studies included application studies, operational analyses, and cost effectiveness studies. The supporting materials screening, fabrication, and testing was included in the technological base activities. These efforts culminated in the actual design, fabrication, and test of component hardware. This culmination presents the component hardware development efforts in the true light as the spearhead of the US Air Force advanced composite materials development program.

1. AIRCRAFT STRUCTURES

The specific aircraft structures component investigated was a high-performance aircraft horizontal stabilizer, seen in Figure 29 (Reference 38). The demonstration item developed was a representative primary load-bearing mid-section of the stabilizer. Two identical items were fabricated. One was for static test and one for fatigue test. The configuration of the items included boron-reinforced-epoxy matrix skins, full depth fiberglass honeycomb core, fiberglass closure spars along each edge, and titanium end fittings. This is a good example of the

materials application philosophy of the entire program which consists of using the best materials from both a cost and performance basis. This philosophy has resulted in a discriminating use of advanced composites only where the high performance of these materials is essential.

Unique test specimens were developed in the technological base activities of the program. Of particular interest were beam specimens constructed of boron-epoxy skins and aluminum-honeycomb core. The laminate configurations used in the skins were comparable to those used in the final skin layups in the two tail components. It was from these beam specimens that the design allowable information was obtained for the computerized design techniques used in the horizontal stabilizer program. The design, fabrication, and test of test specimens with configurations comparable to the end demonstration item is another important philosophy followed in this rapidly progressing technology development.

The static test of the first tail component was extremely successful. The spectrum of loading included both subsonic and supersonic configurations. The aeroelastic response of the component was 101% of design under supersonic conditions and 104% of design under subsonic conditions. The component was tested to destruction which occurred at 133% of design limit load or 89% of design ultimate load for a factor of safety of 1.5. As a result of the subsequent failure analysis, three possible failure modes were postulated. A nonbonded area approximately 1 in. x 6 in. existed between the skin and the titanium end fitting on the compression side of the component. The failure could have been due to compression buckle of the skin which resulted in delamination of the joint. At another area along the failure, stress analysis and strain gage data showed that the load path near one edge of the failure was not as predicted. Consequently, the filament orientation was not optimized and failure could have been initiated in this area. Strain gage data indicated that there was a more rapid transfer of load from the boron-epoxy skins to the titanium end fitting than had been predicted. Thus, this represents the third possible failure mode.

The second tail component was fatigue tested and successfully withstood the planned four lifetimes of the aircraft fatigue spectrum. This component was subsequently failed statically. Failure occurred at 75% of design ultimate load as compared to 89% for the first item. This indicated that only minor damage resulted during the four lifetimes of fatigue.

This horizontal stabilizer program showed that by using the configuration described earlier, a weight savings of 32% is possible. This high payoff and the success of the initial design effort encourage continuing activities in the aircraft structures application area.

2. HELICOPTER ROTOR BLADES

The second systems application area in the initial component development efforts was in the area of helicopter rotor blades. The demonstration items included a 28-inch tail rotor section, main rotor blade root end specimens, and a 6-foot main rotor blade spar. All of the items were designed and applicable to the Bell UH-1F helicopter.

The 28-inch tail rotor section shown in Figure 30 (Reference 38) included boron-epoxy skins, trailing edge and spar, aluminum honeycomb core, and stainless steel leading edge erosion cap. Filament orientation in the skins was ±30 degrees to the span direction, unidirectional in the trailing edge member, and ±10 degrees to the span direction in the spar. The design of the item was intended to attain the same flexure and torsional stiffness as in an all-aluminum counterpart. Testing of the aeroelastic item showed it to be 36% stiffer in flexure, and 41% stiffer in torsion than the aluminum blade section. In addition, the advanced composite blade section was 25% lighter in weight than the aluminum counterpart.

Another major component designed, fabricated, and tested during the program was a 6-ft section of a boron-epoxy main rotor spar illustrated in Figure 31 (Reference 38). The design was a flattened "D" section which matched the upper and lower airfoil contours of the main rotor designed during the initial program. The spar was fatigue tested as a free-free beam. Failure occurred after only 81,400 cycles which was far below the approximately 10^6 cycles anticipated. Subsequent testing of small specimens cut from the spar indicated that the fabrication process had not successfully produced a sound composite material. One specimen ran to destruction at 2,030 cycles and a peak load of 14,500 psi. Another specimen was tested to 8.9×10^6 cycles at a peak loading of 13,000 psi. Investigation of a cross section cut from the spar indicated that the 24-ply-thick structure had not been compacted adequately during the fabrication and curing cycles. Main rotor root end specimens which were fabricated with composite laminates having filament orientations the same as the main rotor spar were successfully fatigue tested to design values.

The success of the aeroelastic tail rotor component and the main rotor blade root end specimens are evidence of the efficient design and fabrication techniques that are available. The premature failure of the main rotor spar section confirms the necessity of producing sound voidfree composite materials which can be depended upon to perform as predicted in design.

Program studies indicate that only marginal performance improvements can be realized by applying advanced composite materials to small helicopter rotor blades. One of the major benefits applicable to these new materials is weight savings. However, weight savings are not necessarily a benefit in the rotor blade application because of the requirement to maintain rotor inertia to a level adequate for auto rotation. Further considerations of applying these materials to other parts of the helicopter system make the weight savings characteristic of continued interest. To design and build a blade of composite materials and take advantage of the ability to tune the frequency of the blade by selective application of high modulus reinforcement materials is perhaps the primary benefit that can be derived in the rotor blade application areas. This benefit cascades into improvements by reducing total system vibration and pilot fatigue, and by increasing the life of the blade.

3. GAS TURBINE ENGINES

The third major area in the initial component effort was that of a gas turbine engine application.

Program studies indicate that considerable weight savings can be realized by using advanced composites in this application area. Using today's conventional materials as a base line, program analyses indicate that a weight savings of 15% in a direct lift engine and 18% in a cruise engine are possible by substitution of organic matrix advanced composites in the cool section of the engines, by using higher temperature organic matrices and advanced metal-matrix composites, a weight savings of 33% in a direct lift engine and 45% in a cruise engine are possible with redesign of the engines.

Three first-stage compressor integral rotor blade and disc assemblies shown in Figure 32 (Reference 38) were fabricated from glass-reinforced epoxy matrix composite material. Two additional rotors were fabricated from a boron-glass epoxy composite. Initial tests on the glass-epoxy rotors were conducted at both vacuum and ambient conditions. Design tip speed was 1500 feet per second. A tip speed of 1220 feet per second was achieved at temperatures exceeding 275°F. Some delamination was noted at the tip and base of the blades. However, structural characteristics of the rotors were such that no damaging vibrational levels were observed. By putting boron reinforcement in the leading edge, trailing edge, and spine of the blades and in the shroud, the delamination problem was reduced significantly. However, the

boron in the blade occupied sufficient space to reduce the amount of glass fibers that were laid continuously through the blades and hub. This reduction of glass through the hub caused the hub to fail prematurely. Testing is continuing on these items.

Sixteen boron-glass-epoxy fan blades were fabricated during the program. Frequency tests showed that the boron composite blade was superior in stiffness to a titanium blade of similar design. It was 50% lighter than the currently used solid titanium blade which weighs 400 grams. Attachment failures showed the need to design an attachment suitable for composites to replace the currently used titanium-blade dovetail configuration. Subsequent work will include additional dovetail pull tests and analysis of redesign attachment configurations. Metal-matrix composites have the potential of providing many solutions to such attachment problems.

Ten stator vanes were designed, fabricated, and tested to demonstrate needed operational stiffness and fatigue properties. The boron-glass-epoxy vanes were tested for fatigue and demonstrated that they were capable of operating indefinitely in the experimental direct-lift engine at engine design loads. Ballistic impact tests were run on the leading and trailing edges of the vanes. Although damaged, the vanes were still three times stiffer than an undamaged fiberglass vane counterpart. The boron-glass vanes also maintained a higher frequency response than the all-glass vanes. No comparisons were made with metal vanes of a similar geometry.

Although these hardware items have shown that considerable payoffs can be achieved by using advanced composites in gas turbine engines, in order to realize the full potential much remains to be done particularly in the high temperature matrix areas. Metal-matrix-composite technology is really in its infancy, but it promises to provide the higher temperature capability and has the potential for application in the attachment problem areas.

4. AEROSPACE VEHICLES

The fourth major area in the initial efforts was the application of advanced composite materials in aerospace vehicles. The specific demonstration component illustrated in Figure 33 (Reference 38) was a cyclinder representing the midsection of an aerospace vehicle. The load configuration that a typical aerospace vehicle conical midsection would experience was reduced to representative loads on the three 18-inch diameter, 24-inch long cylinders which were designed, fabricated, and tested in the program.

The basic design of the cylinders included internal stiffeners integrally wound and cured with the cylinder wall. There are three primary advantages of this design and materials combination over an aluminum substructure counterpart: (1) greater internal volume which will permit increased flexibility and size of load, (2) reduced weight which also permits increased load, and (3) improved aerodynamic stability which results from decreasing the weight in the midsection, thus increasing the distance between the center of gravity and the center of pressure.

The primary purpose of the first of the three cylinders was to check out the automated tape winding procedure and equipment. This was the only hardware program that used a fabrication technique other than hand layup. To conserve the relatively expensive boron filament material, this first item was wound using boron and glass. A plaster mandrel was formed and grooves were cut to accommodate the integrally wound stiffeners. One-eighth-inch-wide boron-reinforced epoxy resin preimpregnated tape was first wound into the bottom of the grooves. A lightweight plastic filler strip was then clamped into each groove leaving sufficient space on each side to permit winding of the sidewalls of the stiffeners. After the stiffener sidewalls were wound, the first circumferential wrap was made on the main wall of the cylinder. This was followed by helical windings and final circumferential windings. After curing in the autoclave, the plaster mandrel was removed and the cylinder inspected. The first cylinder

made of glass and boron tape showed considerable stiffener wall voids. Consequently, more precise control of stiffener wall thickness was desirable. Subsequent cylinders were fabricated using a low-melting-point eutectic metal stiffener filler which was melted and removed after the cylinder was cured. This procedure produced excellent stiffeners.

The second cylinder was designed for three loading conditions. The first was axial tension of 56,200 pounds combined with 102 psi external side pressure. The cylinder was tested to at least 75% of design loads in all three cases. It was then subject to axial tension to destruction; this occurred at 59,900 pounds which was 107% of design load. The failure mode was local bending caused by the end fitting which was bonded to the inside of the cylinder wall.

The third cylinder was of a more optimized design for which maximum advantage was taken of the supporting test information acquired from 6-inch diameter cylinders designed, fabricated, and tested during the program. Designing to the same load configuration as used with cylinder number two required fewer stiffeners than were in number three. In addition, the end fitting was designed for and bonded to both the inner and outer surfaces of the cylinder wall to eliminate the introduction of local bending during test. The test sequence on the third cylinder included two runs to 100% of design under each of the three cases described earlier. The cylinder was not tested to destruction.

These initial ring-stiffened cylindrical structures results have been most promising. However, much remains to be done. Problems to be considered include attaching the mid-section to the rest of the aerospace vehicle, attaching the heat shield, and attaching internal subsystems to the substructure. Again metal matrices hold promise to attachment solutions in the aerospace vehicle area.

SECTION VI

SUMMARY

A review of recent progress in composite technology has been presented with primary emphasis on fiber-reinforced metal-matrix composites. A brief discussion of the development of high strength, high modulus, low density filaments was included. Current effort on plastic and ceramic matrix composites has been considered for comparative purposes. Filament-matrix compatibility has been identified as the major limiting factor in high-temperature fabrication and utilization of metal-matrix composites. Some approaches to the solution of this compatibility problem are discussed. The various fabrication methods are discussed and the mechanical behavior of metal-matrix systems is illustrated with a boron-reinforced aluminum composite. The approach taken to expedite the development of advanced composites as practical engineering structures has been emphasized. This approach has been to integrate the efforts of materials engineers, designers, and fabricators into a single team. A similar method will be used in the development of metal-matrix composites.

The team-approach is illustrated in the development of several filament-reinforced plastic matrix aerospace structures. In the area of gas turbine application, there is a need for increased temperature capability through the use of metal-matrix composites. Composite structure development has shown the necessity for automated tape layup techniques. Hand-layup techniques simply will not be satisfactory for timely and cost effective scale-up to operational systems hardware.

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TABLE I

Exploratory Experiments With Molybdenum Fiber-Body 712

Dimensions of Moly Fiber	% Vol. Fiber	E at R.T. (10^6 psi)	Calc. E (10^6 psi)	Modulus of Rupture After 4 cycles	Calc. % Total Stress Assumed by Fiber	Calc. Stress Assumed by 712
0.008 x 1/4 in.	30	40.5	36	27,900 psi	35.0	18,150 psi
0.002 x 1/8 in.	30	35.7	36	30,700 psi	35.0	20,000 psi
0.006 x 1/4 in.	43	36.5	37.5	36,800 psi	48.8	18,900 psi
0.006 x 3 in.	25	35.4	35.9	32,000 psi	29.0	22,735 psi
0.002 x 1/8 in.	4.2	32.2	34.2	1,400 psi	5.24	___*
None	0	33.8		0		
					Average:	19,946 psi

*This value has no meaning due to the extremely low value of modulus of rupture after four thermal cycles.

TABLE II

Weight Comparison of Rocket-Motor Cases (From Litvak et al, Reference 12)

Material	Strength, psi	Density, lb/in^3	Strength/ Density Ratio, in.	Case Weight, lb
Steel alloy..................	200×10^3	0.283	0.71×10^6	536
Titanium alloy...............	160	0.160	1.00	321
E-glass filament.............	350	0.092	3.81	301
S 994 glass filament..........	437	0.088	4.97	254

TABLE III

Filament Properties

Material	Tensile Strength, ksi	Elastic Modulus, psi	Density, lb/in.^{3}	Strength/ Density Ratio, in.
Beryllium	180	45×10^6	0.066	2.73×10^6
Molybdenum	320	48	0.369	0.88
Steel	600	29	0.283	2.12
Tungsten	580	59	0.697	0.83
E-glass	350	10.5	0.092	3.81
Silica	500	10.5	0.079	6.35
Boron	400	55	0.083	4.82
Carbon	200	25	0.060	2.17
Silicon carbide	250	70	0.142	1.76
Alumina whiskers	2000	75	0.143	24.5
Silicon carbide whiskers	1500	123	0.115	13.0

MATERIALS PROPERTIES/DENSITY

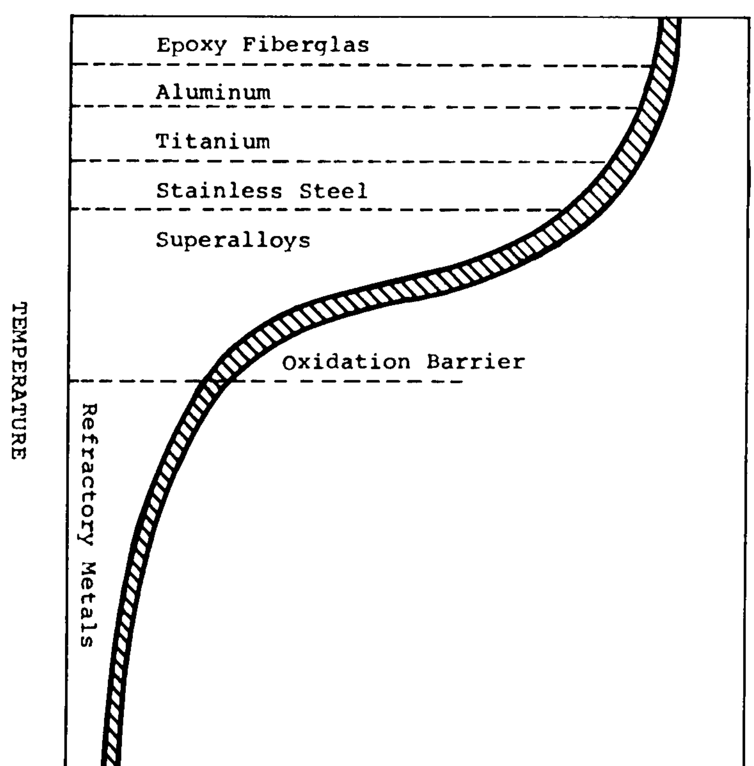


Figure 1. Materials Technology Envelope

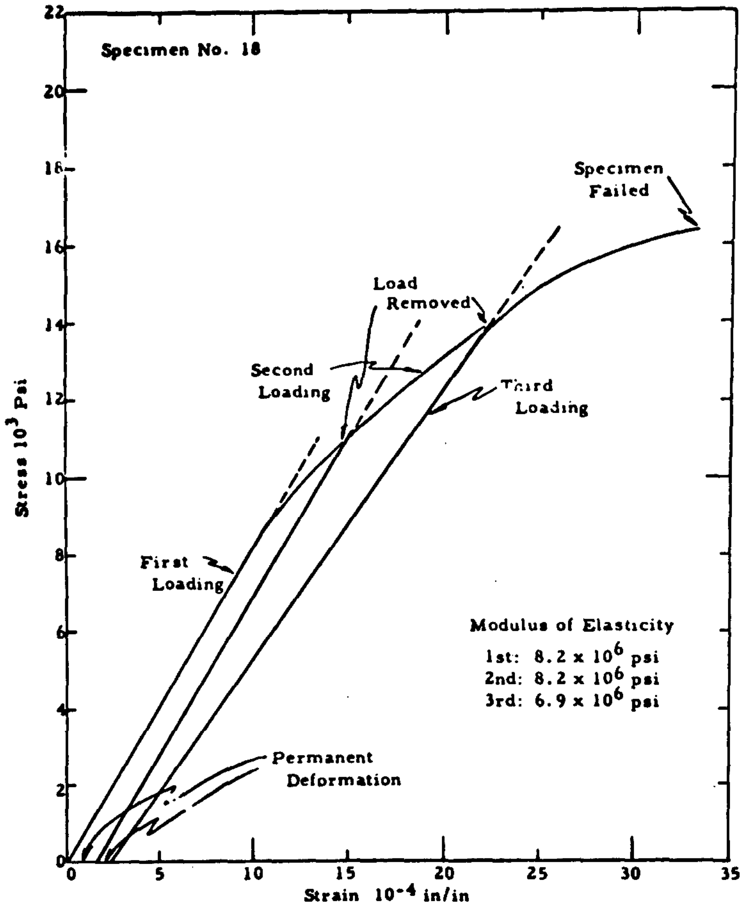


Figure 2. Stress-Strain Diagram For Successive Tests of a 10 Volume Percent Fiber Reinforced Specimen

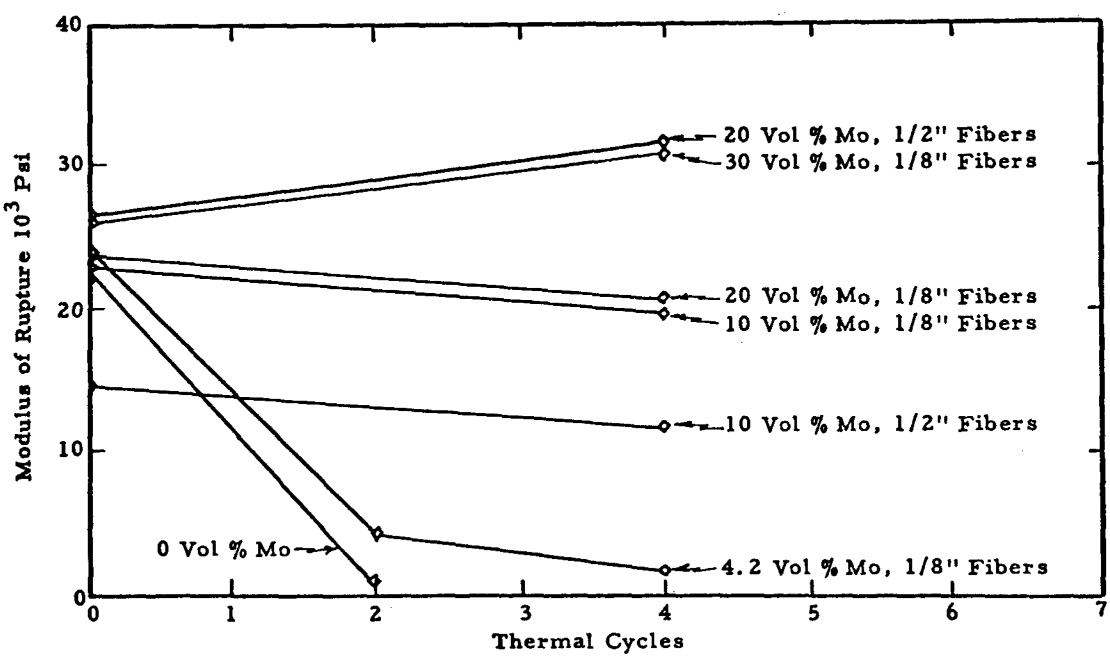


Figure 3. Modulus of Rupture as a Function of Thermal Cycling for the Body 712-.002 Inch Diameter Molybdenum Fiber System

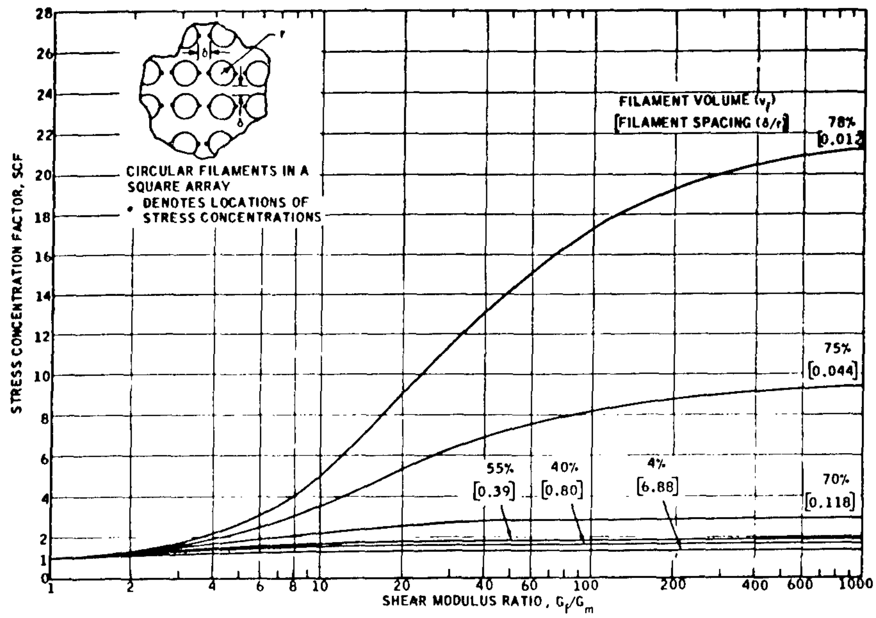


Figure 4. Stress Concentration Factor (SCF) for Circular Filaments in a Square Array Subjected to Longitudinal Shear Loading ($\bar{\tau}_{zx}$)

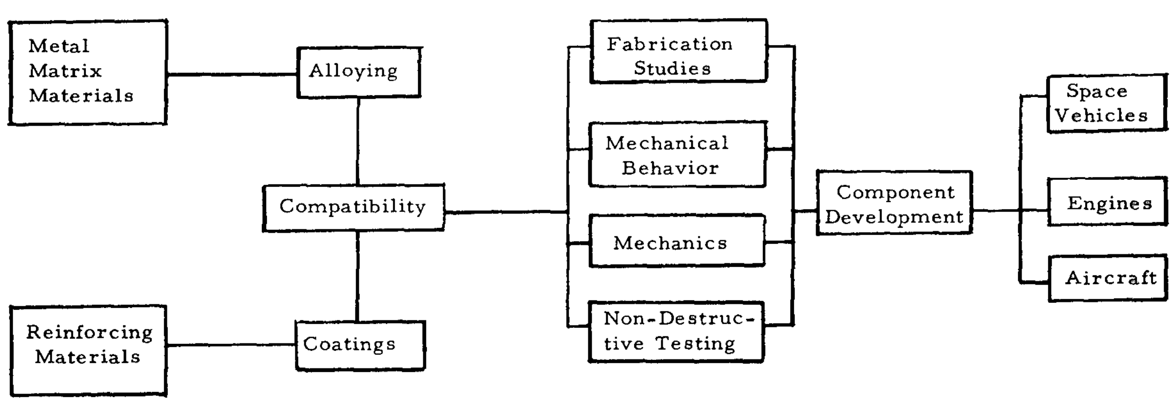


Figure 5. Metal-Matrix Composites

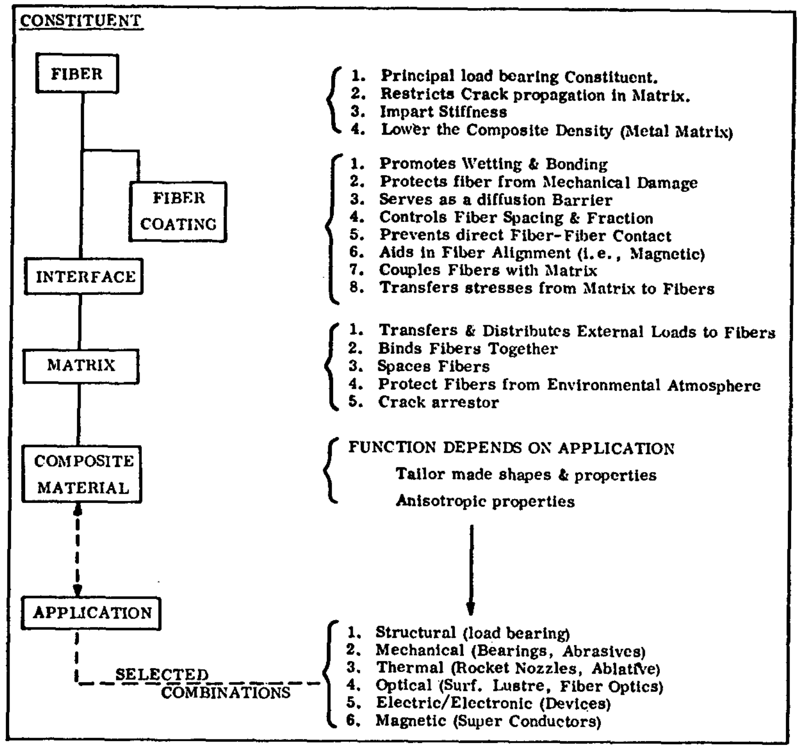


Figure 6. Role of the Constituents in Fiber Composite Materials

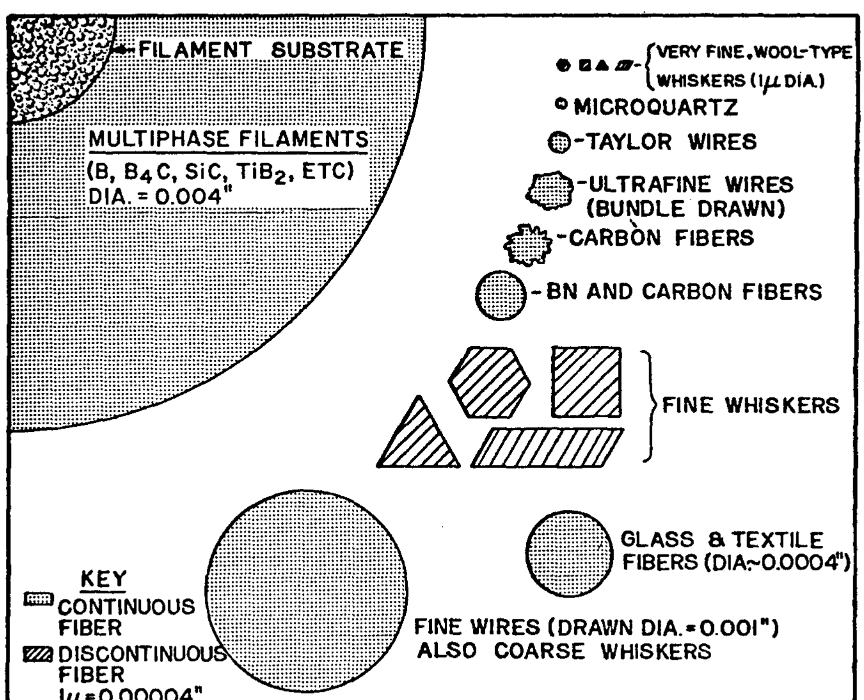


Figure 7. Comparison of the Cross-Sectional Dimensions for Various Inorganic Reinforcements

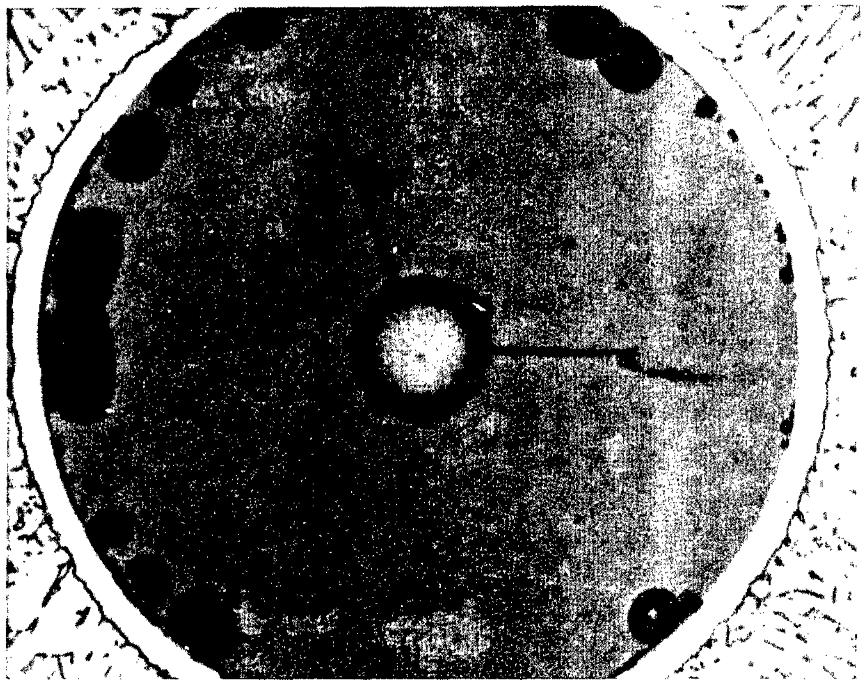


Figure 8. Ti-8Al-1Mo-1V/Boron Composite Showing Void Formation Adjacent to TiB_2/B Interface. (Thermal treatment: 900°C, 10 hours)

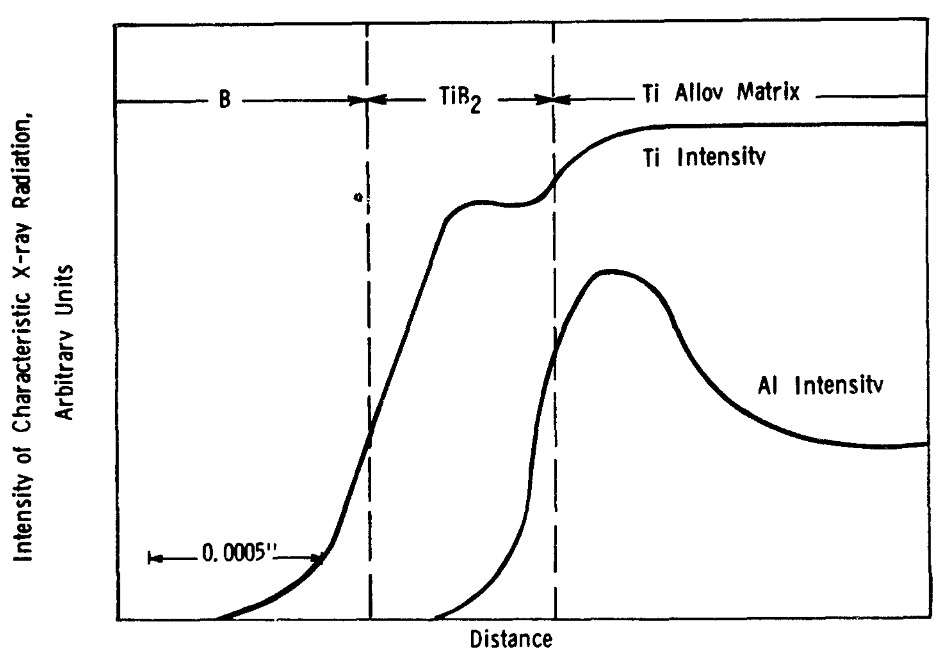


Figure 9. Electron Probe Analyzer Trace Through Reaction Zone in Ti-8Al-1Mo-1V/B Composite Showing Al Rejection from Growing TiB_2 Phase Layer

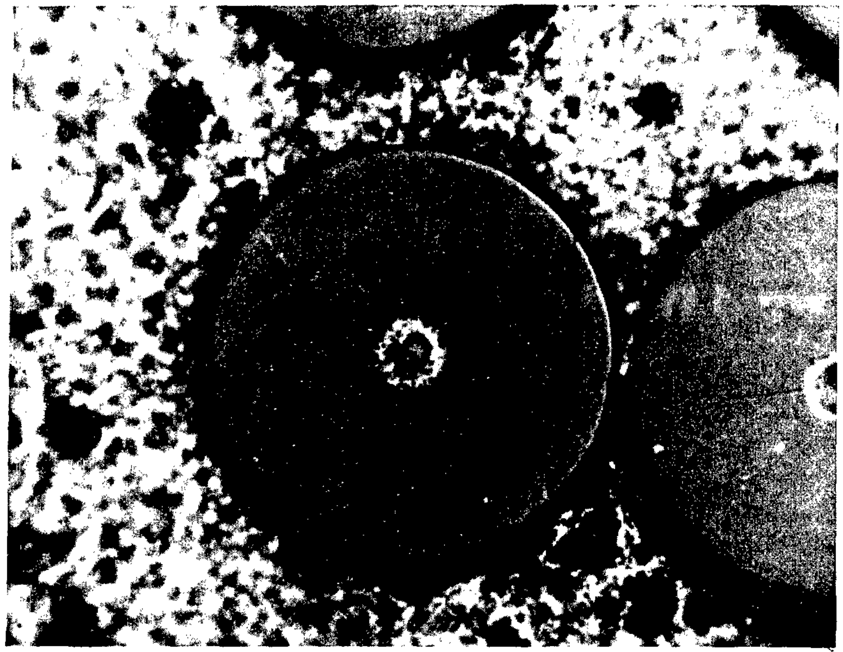


Figure 10. Aluminum/Boron Composite Prepared by Cold-Pressing and Sintering at 625°C for 2 Hours. (No evidence of reaction)

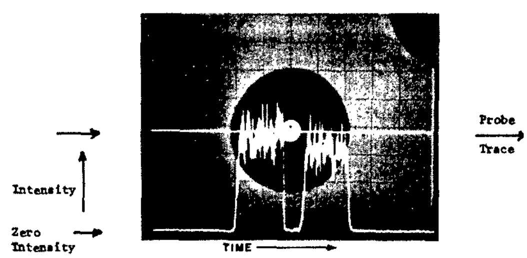


Figure 11(a). Display Showing the Change in Absorbed Specimen Current at 400X and the X-Ray Intensity of Boron. (Composite of boron-reinforced 2024-0 aluminum)

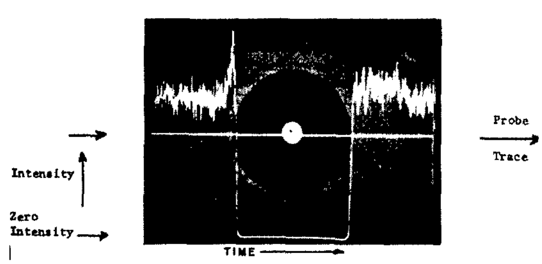


Figure 11(b). Display Showing the Change in Absorbed Specimen Current at 400X and the X-Ray Intensity of Aluminum. (Composite of boron-reinforced 2024-0 aluminum)

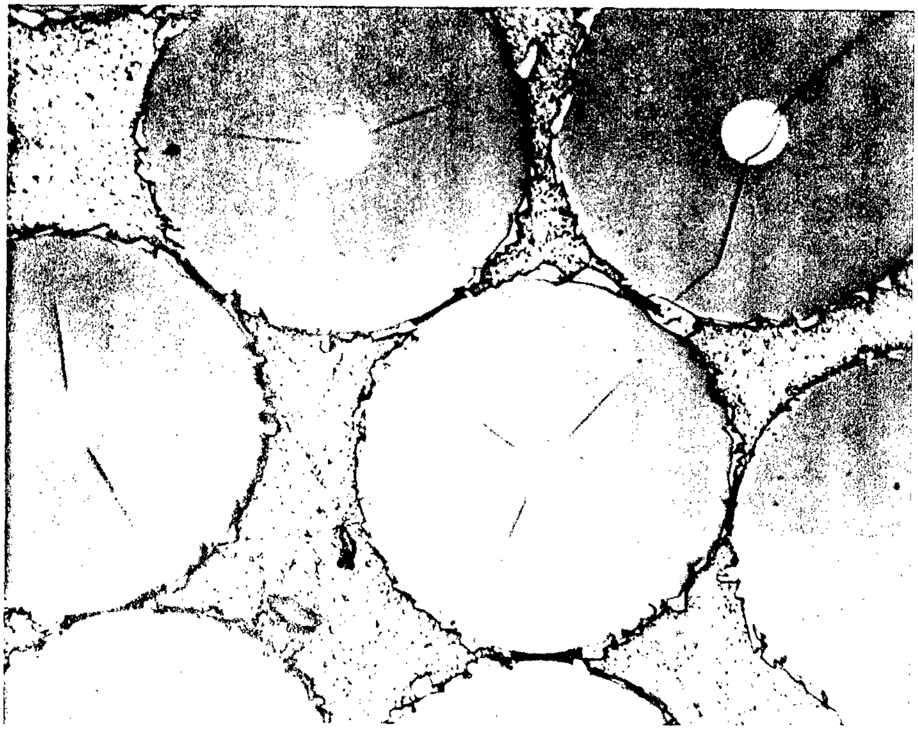


Figure 12. Reaction of Fibers With Aluminum Matrix at 1350°F and 0.5 Minutes

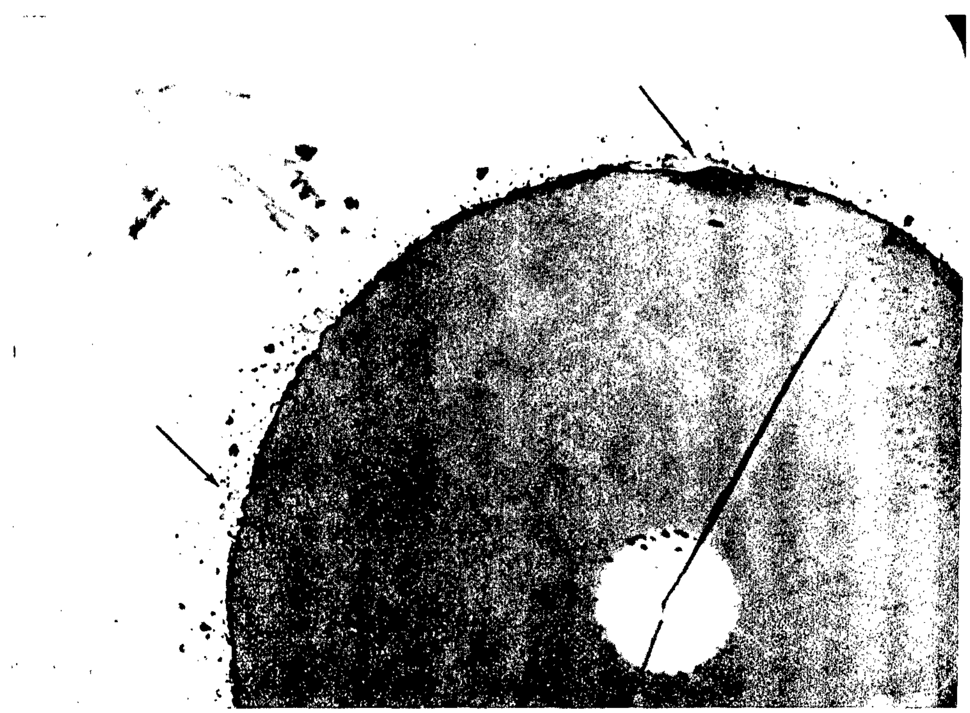


Figure 13. Aluminum-Boron Reaction Product on Aluminum-Coated Boron Fiber (Arrowed). (This reaction product appeared tan and rather indistinct, in contrast to boron chips occasionally found in the aluminum, due to the Polishing Operations, which are gray and well defined)

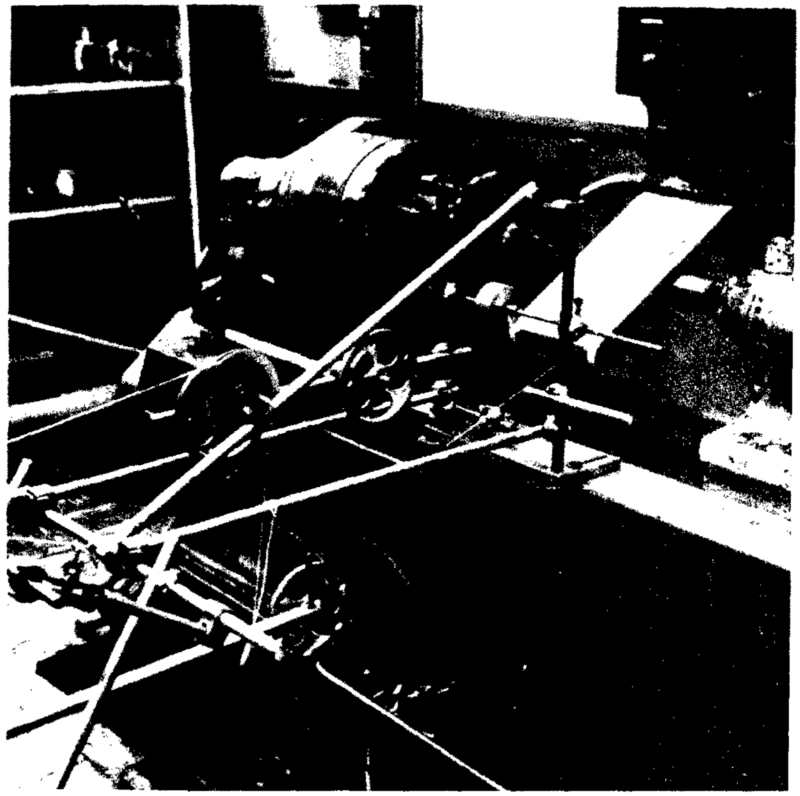


Figure 14. Mandrel Winding Setup

Figure 15. Automated Hand Loom for Weaving Filament Mats

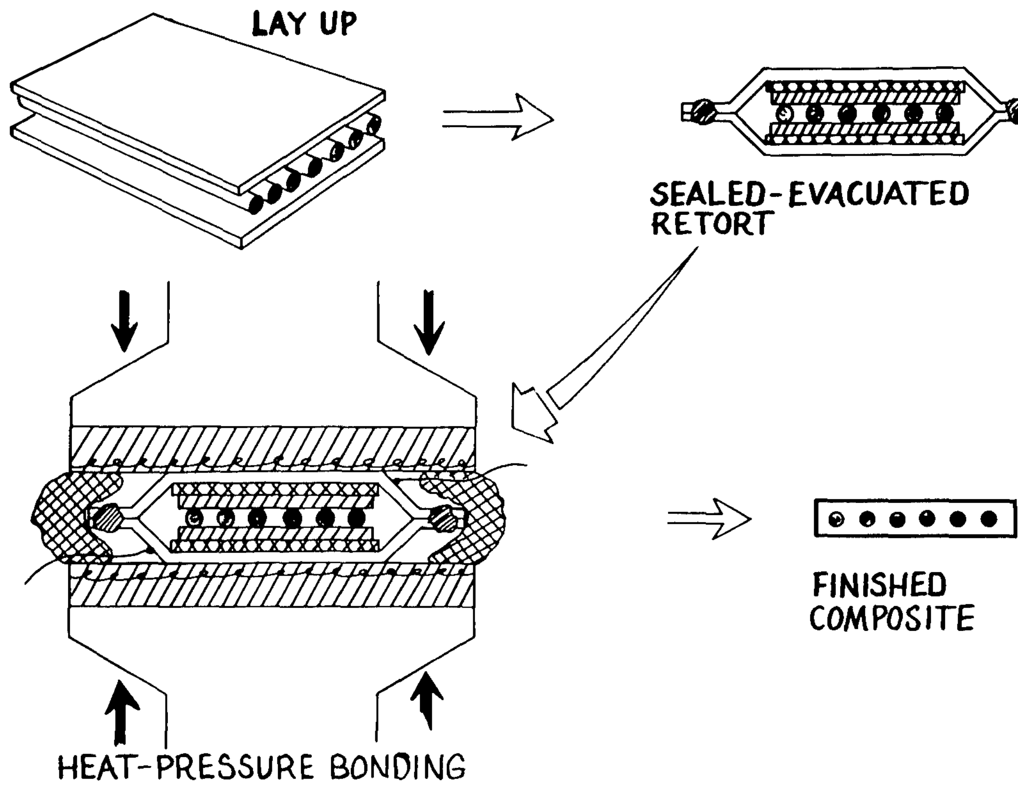


Figure 16. Composite Materials Fabrication Process

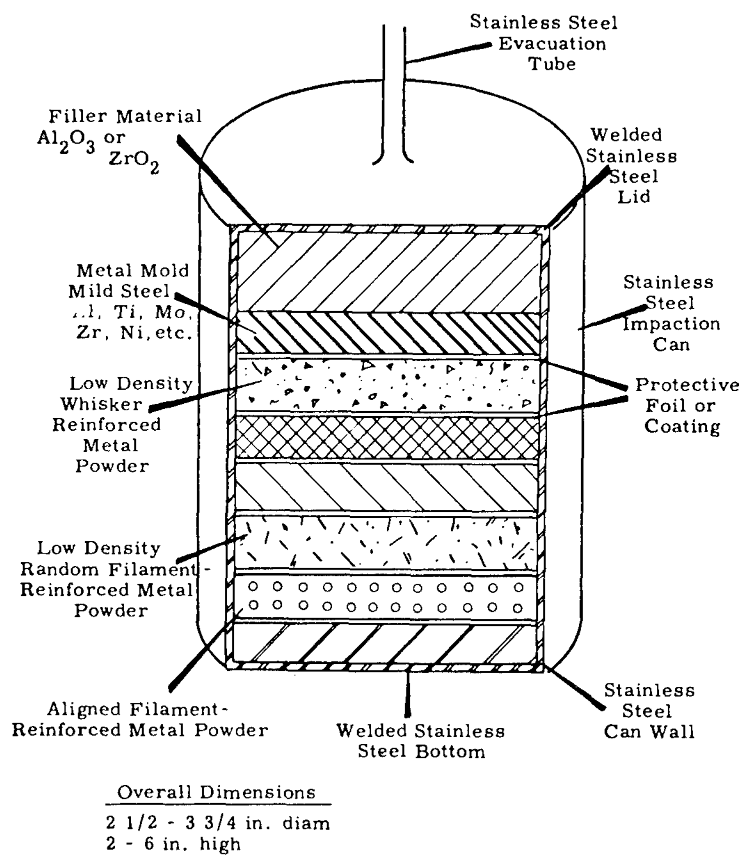


Figure 17. Composite Billet for High Energy-Rate Forming Composite Plates

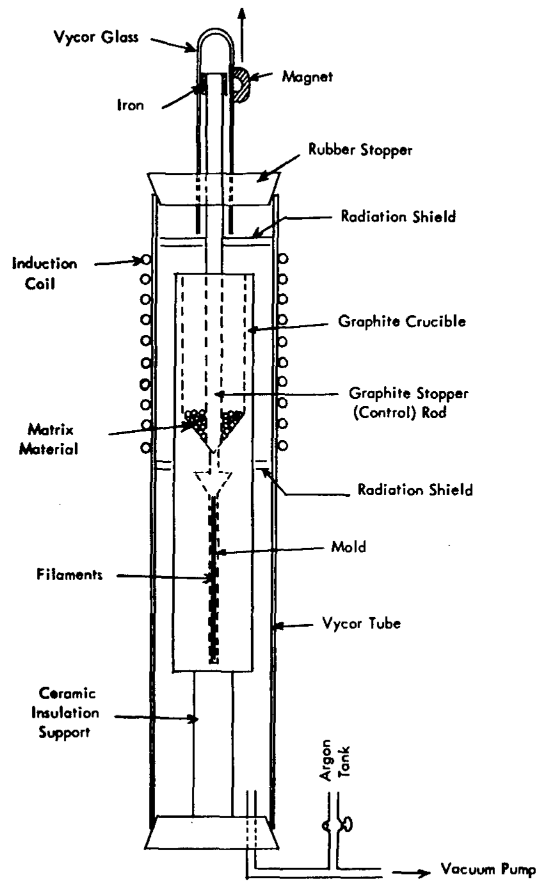


Figure 18. Schematic Diagram of the Apparatus for Vacuum Casting

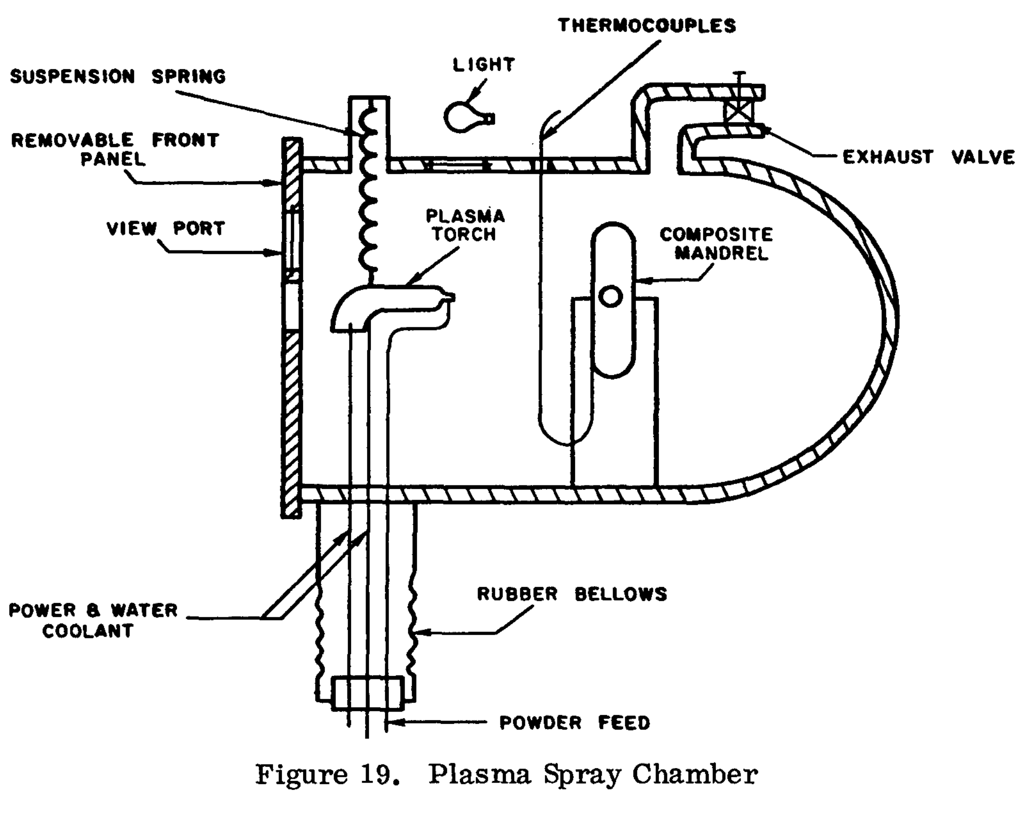


Figure 19. Plasma Spray Chamber

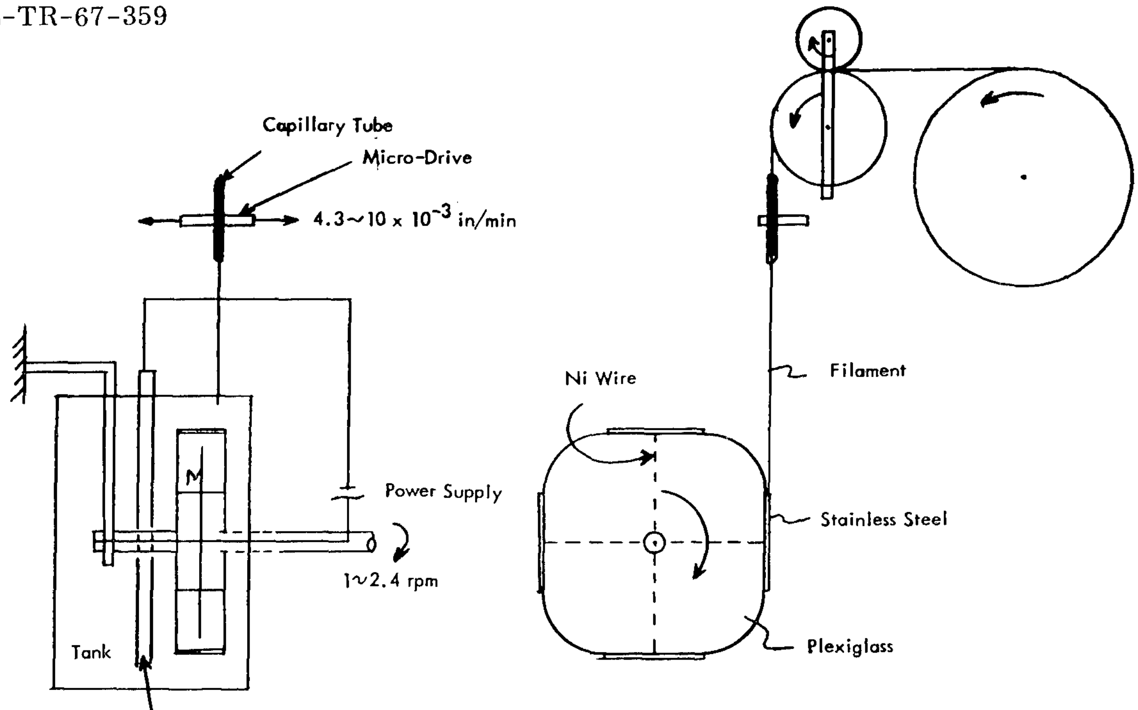
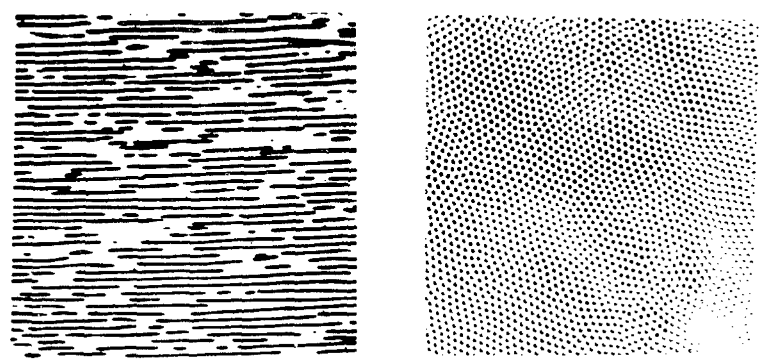
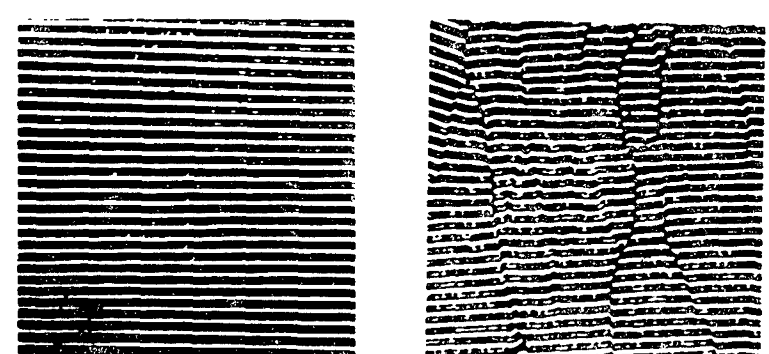


Figure 20. A Schematic Diagram of the Winding Apparatus



Longitudinal Section 200X | Transverse Section 100X

a. $Al\text{-}Al_3Ni$ Exhibiting Rodlike Whiskers



Longitudinal Section 500X | Transverse Section 500X

b. $Al\text{-}CuAl_2$ Exhibiting Lamellar Structure

Figure 21. Microstructure of Unidirectionally Solidified Eutectic

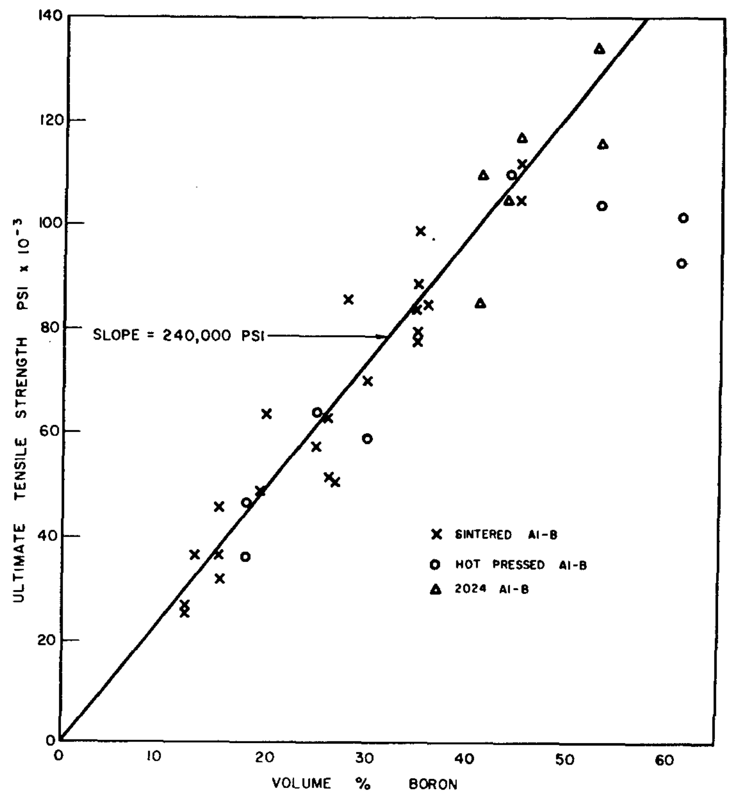


Figure 22. Ultimate Tensile Strength as a Function of Boron Fiber Content

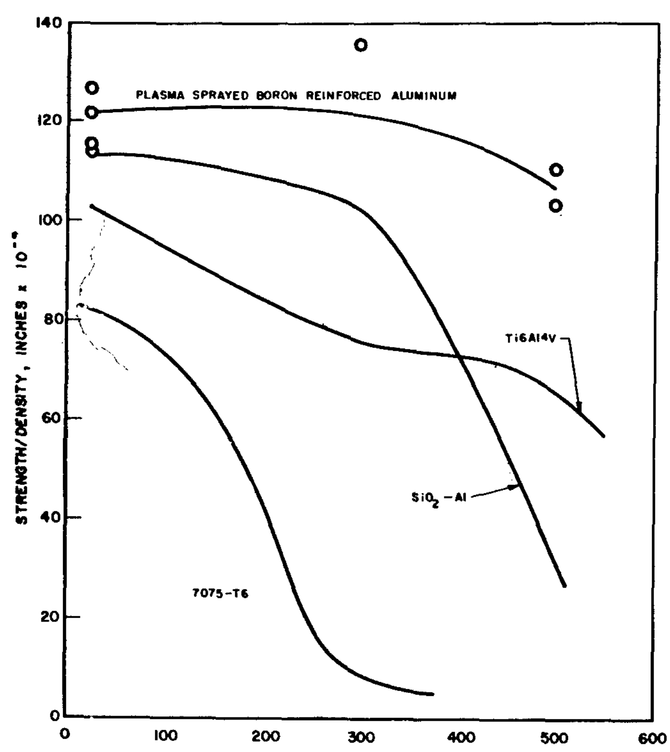


Figure 23. Strength-to-Density Ratio as a Function of Temperature

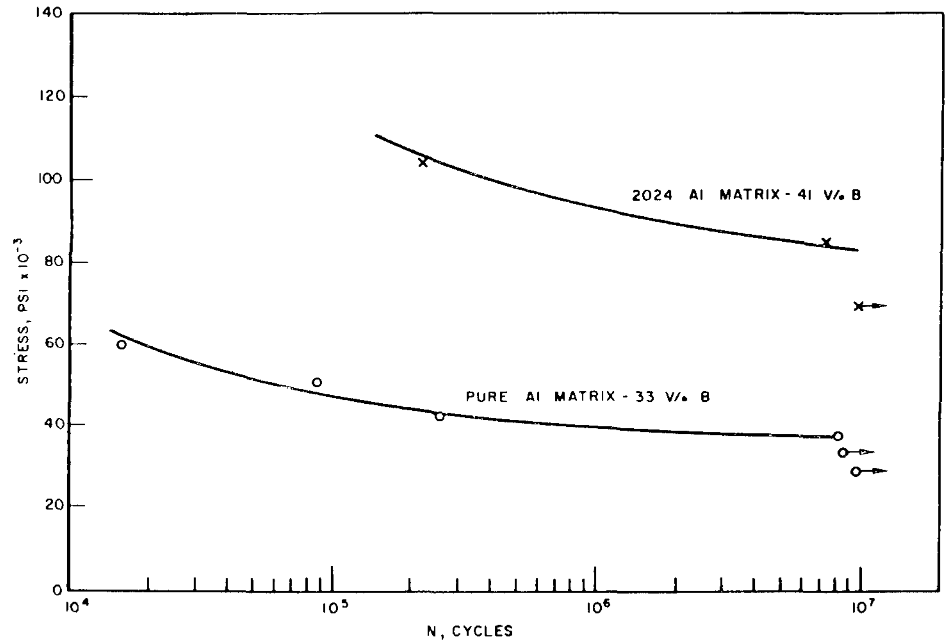


Figure 24. High Cycle Fatigue Behavior of Al-B Composites

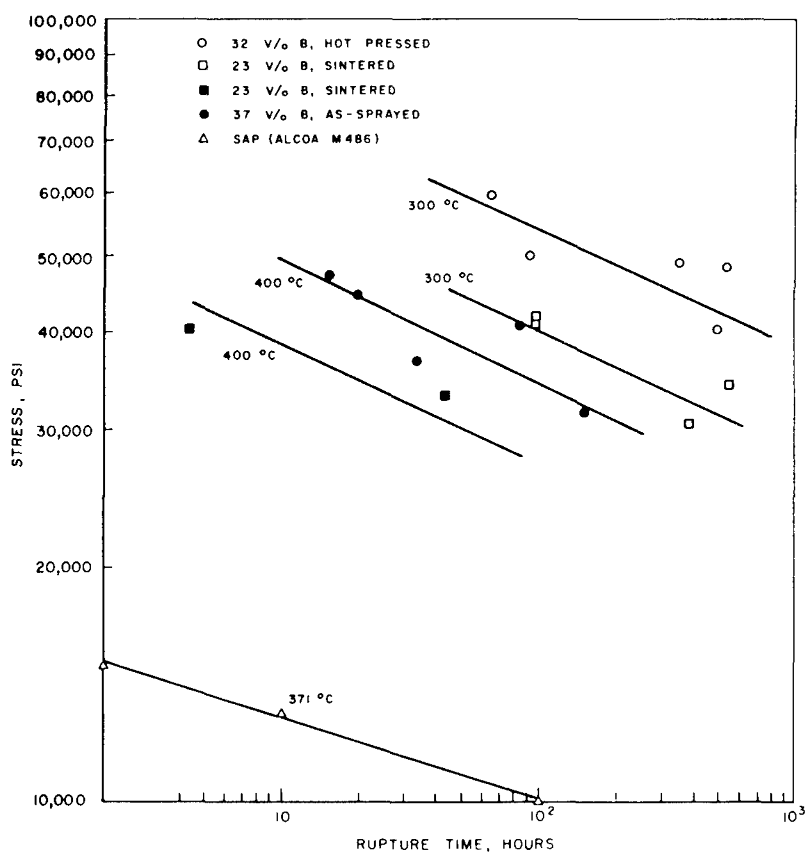


Figure 25. Stress-Rupture Behavior of Boron Fiber-Reinforced Aluminum

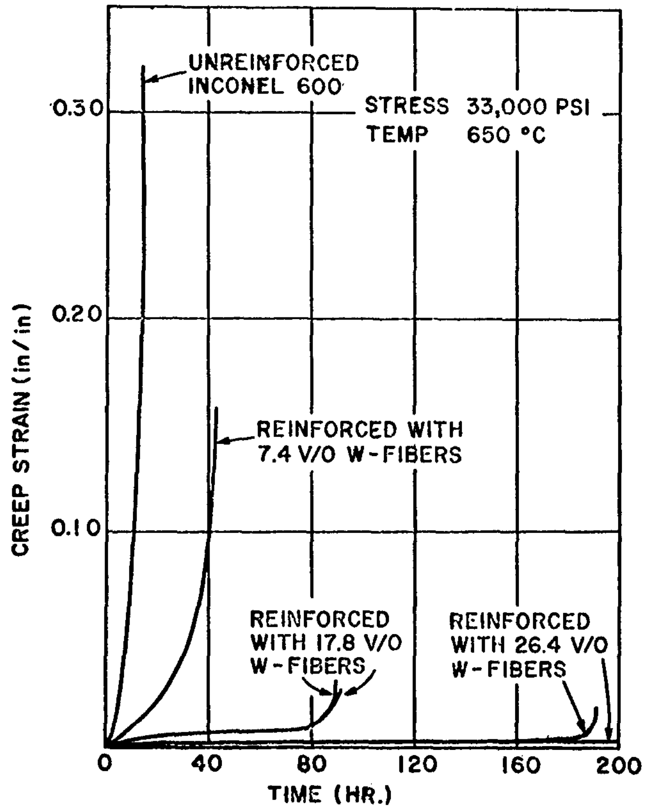


Figure 26. Creep Strain of Inconel 600 Reinforced with Different Volume Fractions of Tungsten Fibers

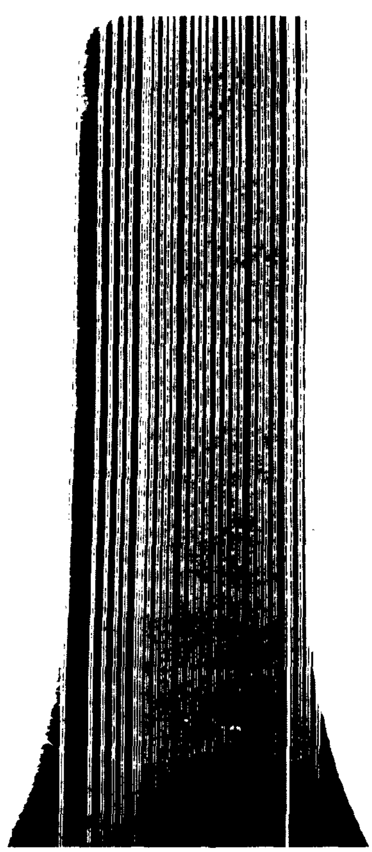


Figure 27. Microradiograph of Boron-Titanium Single Layer Composite, Etched (25KV, 23ma, 2 Hour Exposure)

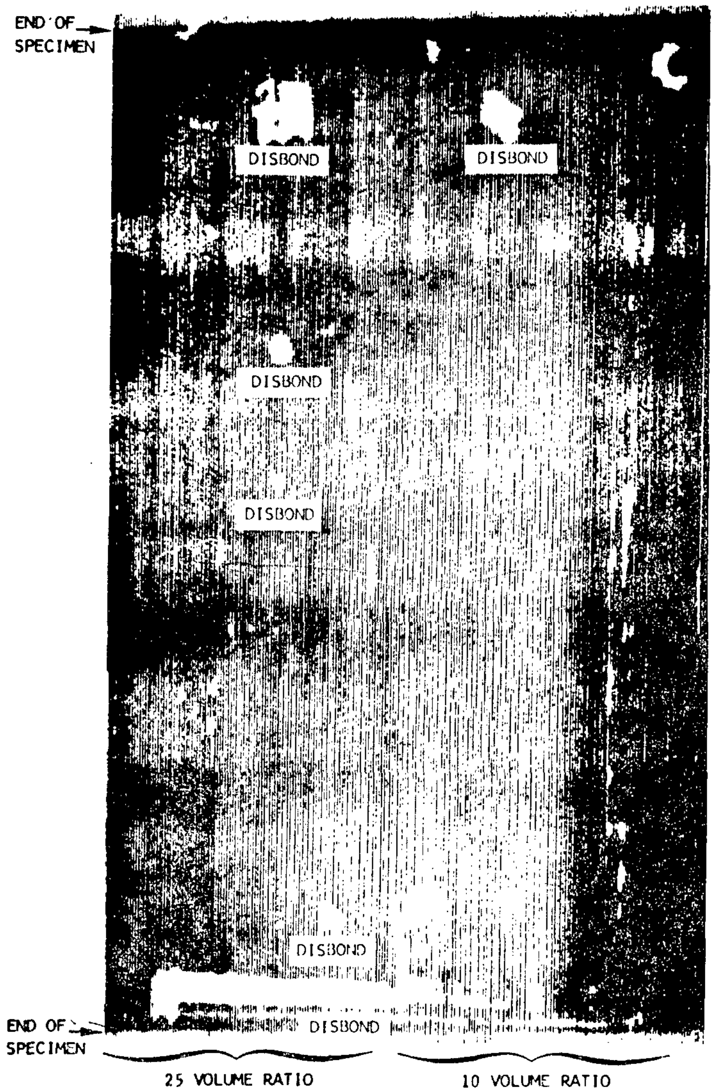


Figure 28. Ultrasonic C-Scan Record of Tungsten-Copper Evaluation Specimen; Test Sensitivity 1.0

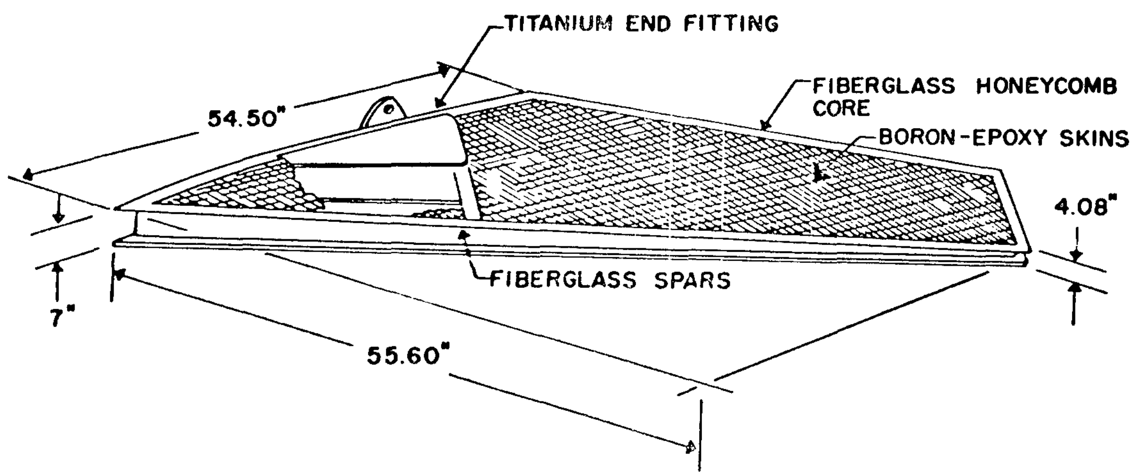


Figure 29. Horizontal Stabilizer Component

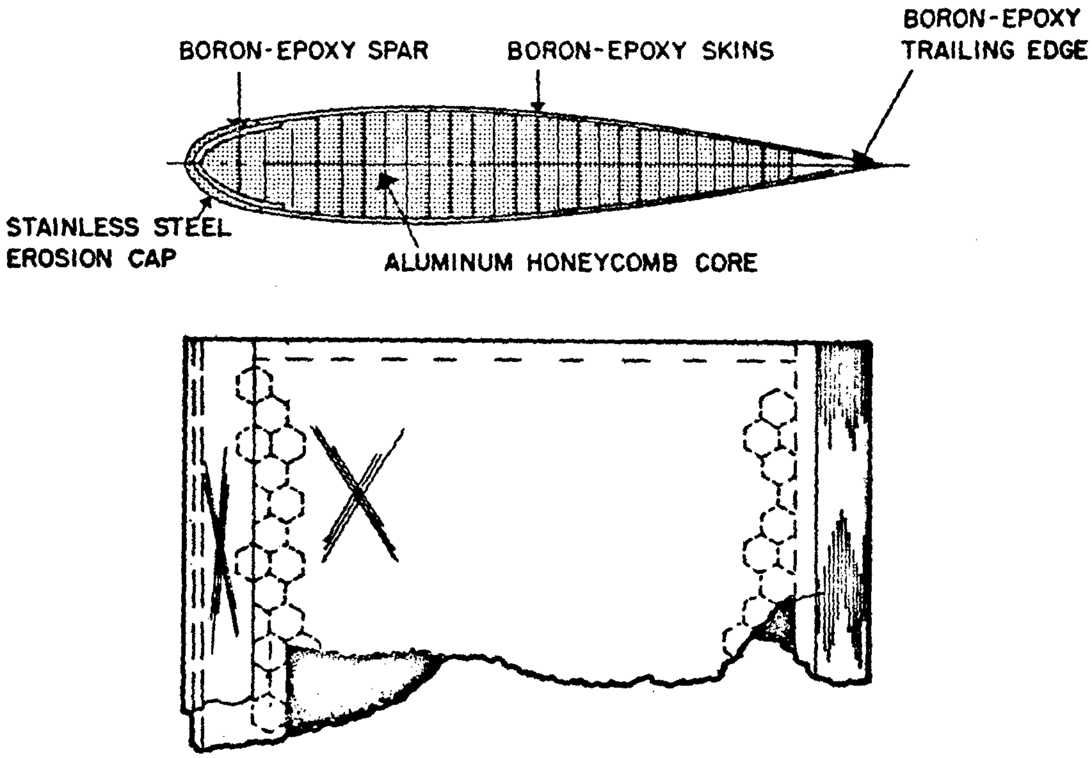


Figure 30. Aeroelastic Tail Rotor Component

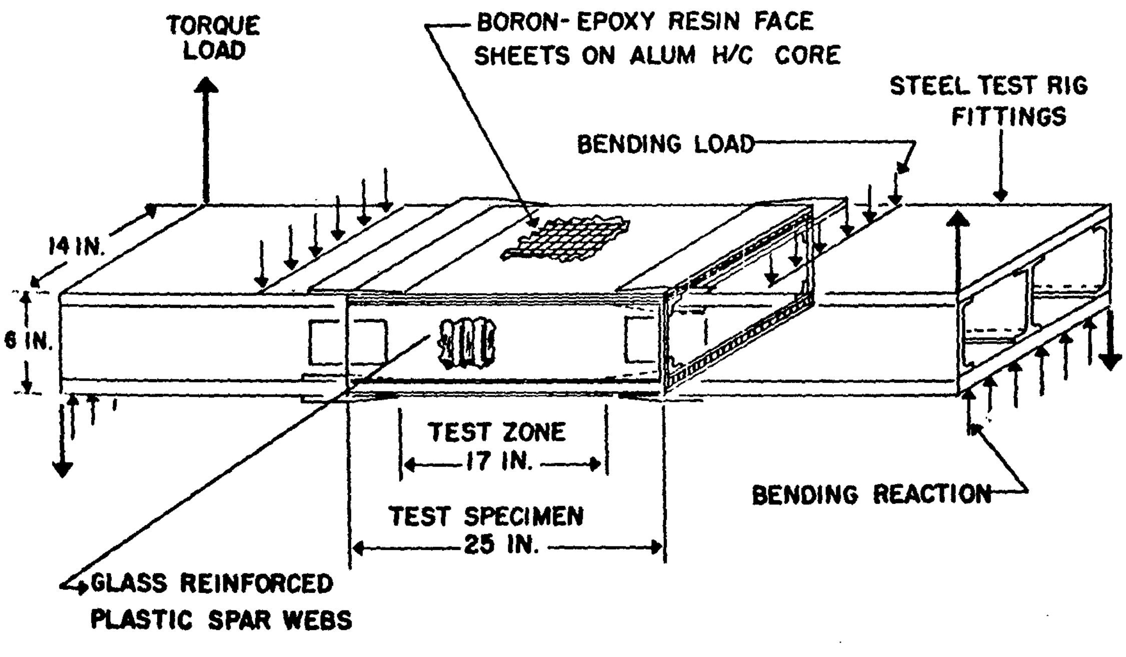


Figure 31. Wing Box Component

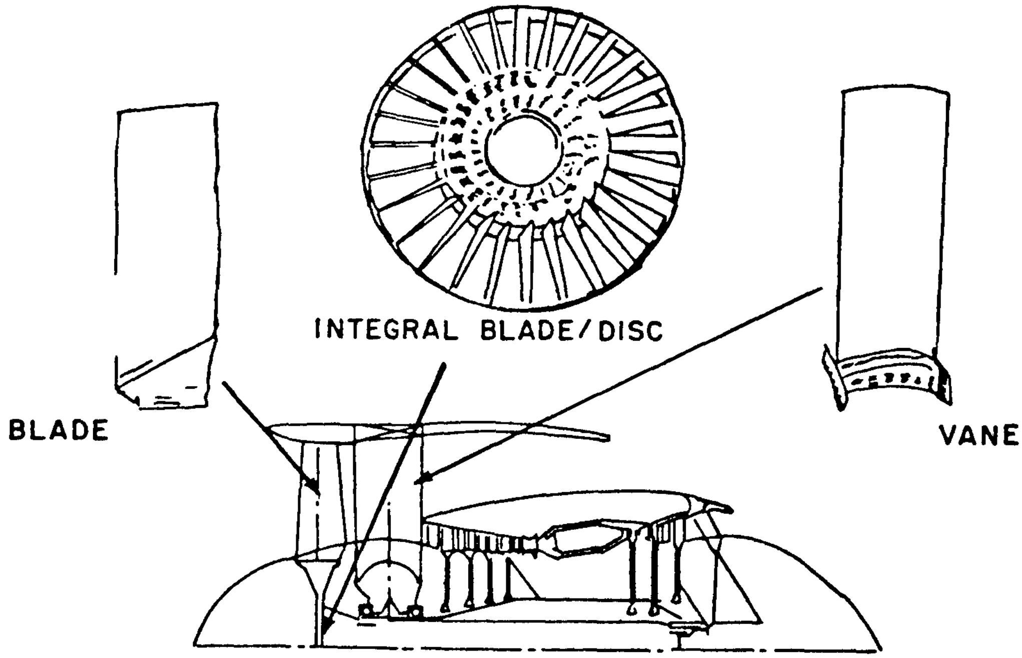


Figure 32. Gas Turbine Engine Components

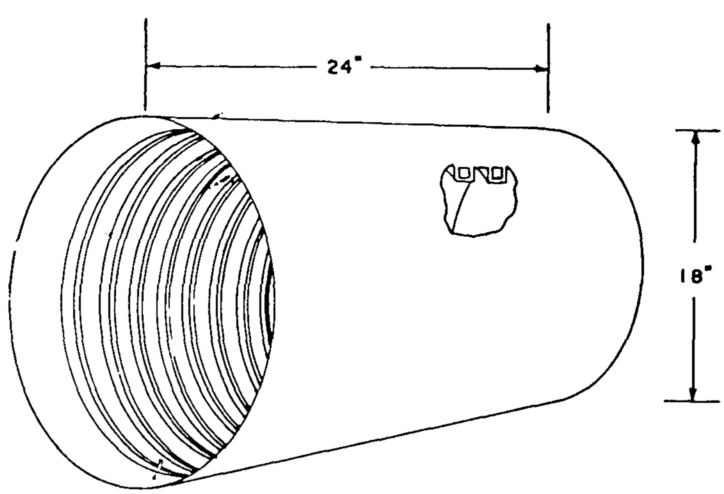


Figure 33. Integrally Ring-Stiffened Cylinder

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13. ABSTRACT

A review of recent progress in composite technology is presented with primary emphasis on fiber-reinforced metal-matrix composites. A brief discussion of the development of high strength, high modulus, low density filaments is included. Current efforts on plastic and ceramic matrix composites have been considered for comparative purposes. Filament-matrix compatibility has been identified as the major limiting factor in high temperature fabrication and utilization of metal-matrix composites. Some approaches to the solution of this compatibility problem are discussed. The various fabrication methods have been reviewed and the mechanical behavior of metal-matrix composites is illustrated with a boron-reinforced aluminum system. The method taken to expedite the development of advanced composites as practical engineering structures is discussed. This approach has been to integrate the efforts of materials engineers, designers, and fabricators into a single team.

This team concept is illustrated by showing the progress of the development of fiber-reinforced plastic aerospace structures. In gas turbine engine applications, the need for increased temperature capability must be met with metal-matrix composites. Fabrication requirements show a definite need for automated tape layup techniques. Hand-layup techniques could not provide cost effective procedures for production of operational systems hardware. Current program efforts indicate that theoretically weight can be reduced by using composite structures. Sometimes in practical applications even greater weight savings can be realized.

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